ATTORNEY DOCKET NO. HOUGE 37-4-33 **PATENT**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

In re Application of: Erik C. Houge, *et al.*

Serial No.: 10/505,197

Filed: June 10, 2005

Title: MONITORING AND CONTROL OF A FABRICATION PROCESS

Grp./A.U.: 1792

Examiner: Robert M. Kunemund Confirmation No.: 3716

Commissioner for Patents
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ATTENTION: Board of Patent Appeals and Interferences

Sirs:

APPEAL BRIEF UNDER 37 C.F.R. §41.37

This is an appeal from a Final Rejection dated December 12, 2007, of Claims 1-21. The Appellants submit this Brief with the statutory fee of $510.00 as set forth in 37 C.F.R.§41.20(b)(2), and hereby authorize the Commissioner to charge any additional fees connected with this communication or credit any overpayment to Deposit Account No. 08-2395.

This Brief contains these items under the following headings, and in the order set forth below in accordance with 37 C.F.R. §41.37(c)(1):

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I. REAL PARTY IN INTEREST

The real party in interest in this appeal is the Assignee, LSI Corporation.

II. RELATED APPEALS AND INTERFERENCES

No other appeals or interferences will directly affect, be directly affected by, or have a bearing on the Board's decision in this appeal.

III. STATUS OF THE CLAIMS

Claims 1-21 are pending in this application and have been rejected under 35 U.S.C. §103(a). Each of the pending claims are being appealed.

IV. STATUS OF THE AMENDMENTS

The present Application with Claims 1-21 was assigned a filing date of June 10, 2005. In the first Examiner's Action electronically delivered June 27, 2007, the Examiner rejected Claims 1-21 under 35 U.S.C. §103(a). The Appellants filed a first Amendment on September 26, 2007, amending Claims 1 and 11. The Examiner considered the first Amendment and subsequently issued a Final Rejection on December 12, 2007, rejecting Claims 1-21 under 35 U.S.C. §103(a). The Appellants filed a Pre-Appeal Brief Request for Review with a Notice of Appeal on March 12, 2008, for Claims 1-21 as filed with the first Amendment of September 26, 2007.

V. SUMMARY OF CLAIMED SUBJECT MATTER

The present invention is directed, in general, to a system and method for controlling a fabrication process and, more specifically, crystallographic metrology and process control for a fabrication process. (*See*, *e.g.*, paragraph 2 of U.S. Patent Application Publication No.2006/0048697, which includes the published specification.) The present invention introduces the broad concept of an inline method and system for monitoring and control of a fabrication process. (*See*, *e.g.*, paragraph 8 of the published specification.)

Independent Claim 1 is directed to a system for monitoring and controlling a fabrication process comprising: (1) a plurality of fabrication subsystems that perform respective fabrication steps on a workpiece; and (2) a crystallographic analysis subsystem for acquiring crystallographic orientation, grain size, or grain morphology from the workpiece after the workpiece undergoes a fabrication step by at least a first subsystem, the crystallographic analysis subsystem coupled to one or more of the fabrication subsystems to provide information for modifying parameters associated with the respective fabrication steps. (*See*, *e.g.*, paragraph 9 of the published specification.)

In one embodiment, a workpiece is first processed through first subsystem 12. The first subsystem 12 may be any known workpiece manufacturing system that lends itself to process control by analysis of the crystallographic properties of the workpiece, such as a semiconductor process or a metal fabrication process. The workpiece is then processed through an analysis subsystem 14 to measure the geometry and quality of the workpiece, such as the crystalline structure of the workpiece. The inline analysis subsystem 14 may include developing a crystallography characterization of the workpiece using atomic force microscopy, surface reflectivity, x-ray diffraction/reflectivity, transmission electron microscopy (TEM), scanning electron microscopy

(SEM) or focused ion beam (FIB) emission. The analysis subsystem 14 may provide a multiple characterization of the workpiece 18 as a feedback signal to the first subsystem, or any other subsystems in the fabrication flow before the analysis subsystem 14. (*See*, *e.g.*, paragraphs 21-22 and Figure 1 of the published specification.)

Independent Claim 11 is directed to a method for monitoring and controlling a fabrication process comprising: (1) coupling a plurality of fabrication subsystems that perform respective fabrication steps on a workpiece; (2) acquiring crystallographic orientation, grain size, or grain morphology from the workpiece after the workpiece undergoes a fabrication step by at least a first subsystem; and (3) providing information to one or more of the fabrication subsystems for modifying parameters associated with the respective fabrication steps.

In one embodiment, for a semiconductor process, fabrication subsystems may include a lithography subsystem and an etch subsystem. The crystallographic orientation, grain size, or grain morphology may be acquired by an inline metrology subsystem that may comprise a scanning electron microscope (SEM), a single focused electron beam (FIB), dual FIBs, or a combination of a SEM/FIB analysis element to determine the quality of the lithography process of the lithography subsystem. After acquiring the crystallographic orientation, grain size, or grain morphology by the inline metrology subsystem, which determines the quality of the lithography process, parameters of the etch process performed by the etch subsystem can be adjusted dynamically to control the process in a feed-forward loop. Similarly, the parameters of the lithography process can be controlled in a feedback loop from the analysis subsystem.

VI. GROUNDS OF REJECTION TO BE REVIEWED ON APPEAL

The issue presented for consideration in this appeal is whether Claims 1-21, as rejected by the Examiner, are patentably nonobvious in accordance with 35 U.S.C. §103(a) over U.S. Patent No. 5,463,977 to Manada, *et al.* ("Manada") in view of U.S. Patent No. 5,466,934 to Adams, *et al.* ("Adams").

VII. APPELLANTS' ARGUMENT

The inventions set forth in independent Claims 1 and 11 and their respective dependent claims are not obvious over the references on which the Examiner relies.

Rejection under 35 U.S.C. 103(a) over Manada in view of Adams

A. Rejection of Claims 1 and 11

The Examiner has rejected Claims 1 and 11 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants respectfully disagree since modifying Manada with Adams renders Manada unsatisfactory for its intended purpose.

In the Final Rejection of December 12, 2007, the Examiner asserts that Manada teaches a subsystem that analyzes crystallographic measurements of a material but that Manada differs from the instant claims in the crystallographic measurements of the material. To cure this deficiency of Manada, the Examiner cites Adams to teach measuring crystallographic properties that Manada does not teach. (*See* Final Rejection of December 12, pages 2-3.) In the Advisory Action of March 7, 2008, the Examiner asserts that Manada teaches away from using a reflective high-energy electron diffraction (RHEED) system and that the system of Adams in not a RHEED system, but another

system which is different. Further, the Examiner asserts the combination does not destroy Manada and that the desire for this art is to increase control over growth and that it would have been obvious to combine Manada and Adams to do so. (*See* Advisory Action of March 7, 2008, Continuation Sheet.)

Manada teaches a method of and an apparatus for epitaxially growing a uniform chemical compound crystal without any defects induced from emission of a high energy electron beam to a surface of the crystal that a conventional RHEED system would create. Manada further teaches placing a chemical compound crystal within a closed chamber of a crystal growing device, alternately introducing a plurality of raw-material gasses into the closed chamber to grow the crystal, emitting a light outside of the closed chamber to a crystal growing film of the crystal inside the closed chamber from a predetermined direction, measuring intensity of the light reflected from the crystal growing film, and controlling charge amounts of the respective raw-material gasses on the basis of a change in the reflected-light intensity, thereby controlling a growing rate of the growing film. (*See, e.g.*, column 1, lines 50-55, column 2, lines 4-14, and Figure 1 of Manada.) Additionally, Manada teaches that the necessity to align a direction of a crystal axis and a direction of the high-energy electron beam with each other in a conventional RHEED system makes it impossible to grow a crystal while rotating a crystal substrate. (*See, e.g.*, column 2, lines 58-62 of Manada.)

Manada overcomes the problems associated with the high-energy electron beam of a conventional RHEED system, namely induced defects, and the related problem of aligning the beam with the direction of a crystal axis by reflecting a light from a light source 1, rather than a high-energy electron beam, off of a growing epitaxial film to help determine the thickness of the growth of an epitaxial layer on a crystal substrate. (*See, e.g.*, column 4, lines 17-19, and Figures 1-3 of

Manada.) Thus, Manada explicitly teaches to avoid using a high-energy electron beam to measure epitaxial thickness so as to prevent introducing defects into a crystalline material and to allow epitaxial crystal growth through rotation of a crystal substrate. As recognized by the Examiner, Manada does not teach acquiring crystallographic orientation, grain size, or grain morphology as is presently claimed.

To cure this recognized deficiency of Manada, the Examiner cites Adams. (*See* Final Rejection of March 7, 2008, page 3.) Adams teaches measuring crystallographic information, specifically identifying crystalline defects, with an imaging apparatus 10 which incorporates a conventional scanning electron microscope (SEM) 12. SEM 12 includes a SEM control unit 14 coupled to an electron beam generator 16 to direct the electron beam generator 16 to discharge a focused electron beam 18 which bombards a material sample 24. (*See, e.g.*, column 2, line 66 through column 3, line 8 and Figure 1 of Adams.) One of ordinary skill in the art at the time of the invention would understand that the electron beam 18 of SEM 12 is a high-energy electron beam. Thus, Adams teaches using a high-energy electron beam to measure crystallographic defects in a material sample. The Examiner states it would have been obvious to one of ordinary skill in the art to modify Manada by the teachings of Adams to measure more than thickness. (*See* Final Rejection of December 12, 2008, page 3.) Even assuming *arguendo* that the SEM 12 of Adams acquires crystallographic orientation, grain size, or grain morphology as presently claimed, Adams explicitly uses a high-energy electron beam to do so. While the system of Adams may not be a conventional RHEED system but another system as the Examiner asserts in the Advisory Action of March 7, 2008, nevertheless Adams explicitly uses a high-energy electron beam.

Manada, while directed to an alternative to a conventional RHEED system, does so to avoid the defects a high energy electron beam used in a conventional RHEED system imparts into a crystalline material. It is Adams' explicit use of a high-energy beam that would render Manada unsatisfactory for its intended purpose since Manada explicitly teaches that the use of a high-energy electron beam, whether from a conventional RHEED system or another system that uses a high-energy electron beam, induces defects into the crystal and does not allow for rotating a crystal substrate to grow an epitaxial layer on it, which Manada is directed to. Modifying Manada with Adams to measure more than just crystal thickness renders Manada unsatisfactory for its intended purpose.

MPEP §2143.01 states that "If proposed modification would render the prior art invention being modified unsatisfactory for it intended purpose, the there is no suggestion or motivation to make the proposed modification." *In re Gordon*, 733 F.2d 900, 221 USPQ 1125(Fed. Cir. 1984). Combining Manada with Adams to modify Manada would do just this, render Manada unsatisfactory for its intended purpose. As such, the cited combination of Manada and Adams, as applied by the Examiner, does not establish a *prima facie* case of obviousness of independent Claims 1 and 11 and Claims that depend thereon. Accordingly, the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 1 and 11 and allow issuance thereof.

B. Rejection of Claims 2 and 12

The Examiner has rejected Claims 2 and 12 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants believe the above arguments establish that the

invention of independent Claims 1 and 11 is not obvious in view of the cited combination of Manada and Adams and, therefore, are incorporated herein by reference. Dependent Claims 2 and 12 additionally require providing, in a feed back path, information generated in the crystallographic analysis subsystem to at least the first subsystem. The cited combination of Manada and Adams, however, does not teach or suggest providing, in a feed back path, information generated in the crystallographic analysis subsystem to at least the first subsystem in combination with the base claim limitations. Accordingly, Claims 2 and 12 are nonobvious over the cited combination of Manada and Adams and the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 2 and 12 and allow issuance thereof.

C. Rejection of Claims 3 and 13

The Examiner has rejected Claims 3 and 13 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants believe the above arguments in section A establish that the invention of independent Claims 1 and 11 is not obvious in view of the cited combination of Manada and Adams and, therefore, are incorporated herein by reference. Dependent Claims 3 and 13 additionally require providing, in a feed forward path, information generated in the crystallographic analysis subsystem to at least a second subsystem that performs a fabrication step after the first subsystem and after crystallographic analysis of the workpiece. The cited combination of Manada and Adams, however, does not teach or suggest providing, in a feed forward path, information generated in the crystallographic analysis subsystem to at least a second subsystem that performs a fabrication step after the first subsystem and after crystallographic analysis of the workpiece in combination with the base claim limitations. Accordingly, Claims 3 and 13 are nonobvious over the

cited combination of Manada and Adams and the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 3 and 13 and allow issuance thereof.

D. Rejection of Claims 4, 14, and 15

The Examiner has rejected Claims 4, 14, and 15 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants believe the above arguments in section A establish that the invention of independent Claims 1 and 11 is not obvious in view of the cited combination of Manada and Adams and, therefore, are incorporated herein by reference. Dependent Claims 4 and 14 additionally require characterizing a roughness of a workpiece and dependent Claim 15 further measures a reflectivity in different regions of the workpiece and generates a roughness characteristic based on the measured reflectivity, and thereby introduce a patently distinct limitation in addition to the limitation recited in Claims 1 and 11. The cited combination of Manada and Adams, however, does not teach or suggest characterizing a roughness of a workpiece and further measuring a reflectivity in different regions of the workpiece to generate a roughness characteristic based on the measured reflectivity in combination with the base claims. Thus, the cited combination of Manada and Adams does not establish a *prima facie* case of obviousness of dependent Claims 4, 14, and 15. Accordingly, Claims 4, 14, and 15 are nonobvious over the cited combination of Manada and Adams and the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 4, 14, and 15 and allow issuance thereof.

E. Rejection of Claims 5 and 16

The Examiner has rejected Claims 5 and 16 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants believe the above arguments in section A establish that the invention of independent Claims 1 and 11 is not obvious in view of the cited combination of Manada and Adams and, therefore, are incorporated herein by reference. Dependent Claims 5 and 16 additionally require characterizing a reflectivity of a workpiece. The cited combination of Manada and Adams, however, does not teach or suggest characterizing a reflectivity of a workpiece in combination with the base claim limitations. Accordingly, Claims 5 and 16 are nonobvious over the cited combination of Manada and Adams and the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 5 and 16 and allow issuance thereof.

F. Rejection of Claims 6 and 17

The Examiner has rejected Claims 6 and 17 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants believe the above arguments in section A establish that the invention of independent Claims 1 and 11 is not obvious in view of the cited combination of Manada and Adams and, therefore, are incorporated herein by reference. Dependent Claims 6 and 17 additionally require characterizing a crystallography of a workpiece. The cited combination of Manada and Adams, however, does not teach or suggest characterizing a crystallography of a workpiece in combination with the base claim limitations. Accordingly, Claims 6 and 17 are nonobvious over the cited combination of Manada and Adams and the Appellants respectfully

request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 6 and 17 and allow issuance thereof.

G. Rejection of Claims 7 and 18

The Examiner has rejected Claims 7 and 18 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants believe the above arguments in section A establish that the invention of independent Claims 1 and 11 is not obvious in view of the cited combination of Manada and Adams and, therefore, are incorporated herein by reference. Dependent Claims 7 and 18 additionally require: (1) providing a workpiece holder for holding the workpiece for characterization of a workpiece area; (2) generating an electron beam; (3) controlling the relative movement between the electron beam and the workpiece for directing the electron beam at a series of spaced apart points within the workpiece area; (4) generating crystallographic data based upon electron diffraction from the workpiece; (5) determining whether sufficient data have been acquired to characterize the workpiece area; and, (6) controlling a scanning actuator to space the points apart such that acquired data is representative of a different grain with the workpiece. The cited combination of Manada and Adams, however, does not teach or suggest: (1) providing a workpiece holder for holding the workpiece for characterization of a workpiece area; (2) generating an electron beam; (3) controlling the relative movement between the electron beam and the workpiece for directing the electron beam at a series of spaced apart points within the workpiece area; (4) generating crystallographic data based upon electron diffraction from the workpiece; (5) determining whether sufficient data have been acquired to characterize the workpiece area; and, (6) controlling a scanning actuator to space the points apart such that acquired data is representative of a different grain with the workpiece in

combination with the base and intervening claim limitations. Accordingly, Claims 7 and 18 are nonobvious over the cited combination of Manada and Adams and the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 7 and 18 and allow issuance thereof.

H. Rejection of Claims 8 and 19

The Examiner has rejected Claims 8 and 19 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants believe the above arguments in section A establish that the invention of independent Claims 1 and 11 is not obvious in view of the cited combination of Manada and Adams and, therefore, are incorporated herein by reference. Dependent Claims 8 and 19 additionally require: (1) providing a workpiece holder for holding a crystalline workpiece; (2) generating a first ion beam; (3) controlling the relative movement between the first ion beam and the crystalline workpiece for directing the first ion beam at desired areas of the crystalline workpiece; (4) detecting secondary electrons emitted from the crystalline workpiece; (5) creating a contrast intensity image based upon secondary electron emissions from the crystalline workpiece; (6) providing crystallographic information based on the contrast image intensity data; and, (7) controlling a scanning actuator for scanning the first ion beam. The cited combination of Manada and Adams, however, does not teach or suggest: (1) providing a workpiece holder for holding a crystalline workpiece; (2) generating a first ion beam; (3) controlling the relative movement between the first ion beam and the crystalline workpiece for directing the first ion beam at desired areas of the crystalline workpiece; (4) detecting secondary electrons emitted from the crystalline workpiece; (5) creating a contrast intensity image based upon secondary electron emissions from the crystalline

workpiece; (6) providing crystallographic information based on the contrast image intensity data; and, (7) controlling a scanning actuator for scanning the first ion beam in combination with the base and intervening claim limitations. Accordingly, Claims 8 and 19 are nonobvious over the cited combination of Manada and Adams and the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 8 and 19 and allow issuance thereof.

I. Rejection of Claims 9 and 20

The Examiner has rejected Claims 9 and 20 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants believe the above arguments in section A establish that the invention of independent Claims 1 and 11 is not obvious in view of the cited combination of Manada and Adams and, therefore, are incorporated herein by reference. Dependent Claims 9 and 20 additionally require generating a second ion beam and controlling the relative movement between the first ion beam, the second ion beam, and the crystalline workpiece for directing the second ion beam at desired areas of the crystalline workpiece. The cited combination of Manada and Adams, however, does not teach or suggest generating a second ion beam and controlling the relative movement between the first ion beam, the second ion beam, and the crystalline workpiece for directing the second ion beam at desired areas of the crystalline workpiece in combination with the base and intervening claim limitations. Accordingly, Claims 9 and 20 are nonobvious over the cited combination of Manada and Adams and the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 9 and 20 and allow issuance thereof.

J. Rejection of Claims 10 and 21

The Examiner has rejected Claims 10 and 21 under 35 U.S.C. §103(a) as being unpatentable over Manada in view of Adams. The Appellants believe the above arguments in section A establish that the invention of independent Claims 1 and 11 is not obvious in view of the cited combination of Manada and Adams and, therefore, are incorporated herein by reference. Dependent Claims 10 and 21 additionally require: (1) providing a sample holder for holding a crystalline sample; (2) generating a first ion beam; (3) generating an electron beam; (4) controlling the relative movement between the first ion beam, the electron beam, and the crystalline sample; (5) a scanning actuator being controllable for directing the first ion beam at desired areas of the crystalline sample and for directing the electron beam at a series of points within the sample area; (6) detecting secondary electron emissions from the crystalline sample; (7) creating a contrast intensity image based upon secondary electron emissions from the crystalline sample and generating crystallographic data based upon electron diffraction from the crystalline sample; (8) providing crystallographic information based on the contrast image intensity data and configured for determining whether sufficient data have been acquired to characterize the sample area; and, (9) controlling the scanning actuator to direct the first ion beam at desired areas such that each ion channeling image is representative of channeling directions within the crystalline sample and to space the points apart such that acquired data is representative of a different grain within the crystalline sample.

The cited combination of Manada and Adams, however, does not teach or suggest: (1) providing a sample holder for holding a crystalline sample; (2) generating a first ion beam; (3) generating an electron beam; (4) controlling the relative movement between the first ion beam, the electron beam, and the crystalline sample; (5) a scanning actuator being controllable for directing the

first ion beam at desired areas of the crystalline sample and for directing the electron beam at a series of points within the sample area; (6) detecting secondary electron emissions from the crystalline sample; (7) creating a contrast intensity image based upon secondary electron emissions from the crystalline sample and generating crystallographic data based upon electron diffraction from the crystalline sample; (8) providing crystallographic information based on the contrast image intensity data and configured for determining whether sufficient data have been acquired to characterize the sample area; and, (9) controlling the scanning actuator to direct the first ion beam at desired areas such that each ion channeling image is representative of channeling directions within the crystalline sample and to space the points apart such that acquired data is representative of a different grain within the crystalline sample in combination with the base and intervening claim limitations. Accordingly, Claims 10 and 21 are nonobvious over the cited combination of Manada and Adams and the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of Claims 10 and 21 and allow issuance thereof.

For the reasons set forth above, the Claims on appeal are patentably nonobvious over Manada and Adams. Accordingly, the Appellants respectfully request that the Board of Patent Appeals and Interferences reverse the Examiner's Final Rejection of all of the Appellants' pending claims.

Respectfully submitted,

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VIII. APPENDIX A - CLAIMS

1. A system for monitoring and controlling a fabrication process comprising:

a plurality of fabrication subsystems that perform respective fabrication steps on a workpiece; and

a crystallographic analysis subsystem for acquiring crystallographic orientation, grain size, or grain morphology from the workpiece after the workpiece undergoes a fabrication step by at least a first subsystem, the crystallographic analysis subsystem coupled to one or more of the fabrication subsystems to provide information for modifying parameters associated with the respective fabrication steps.

2. The system of Claim 1, further comprising a feedback path from the crystallographic analysis subsystem to at least the first subsystem for providing information to at least the first subsystem.

3. The system of Claim 1, further comprising a feed-forward path from the crystallographic analysis subsystem to at least a second subsystem that performs a fabrication step after the first subsystem and after crystallographic analysis of the workpiece, the feed forward path providing information to at least the second subsystem.

4. The system of Claim 1, the analysis subsystem further comprising a workpiece roughness characterization subsystem.

5. The system of Claim 1, the analysis subsystem further comprising a workpiece reflectivity characterization subsystem.

6. The system of Claim 1, the analysis subsystem further comprising a crystallographic characterization subsystem.

7. The system of claim 6, the crystallographic characterization system further comprising:

a workpiece holder for holding the workpiece for characterization of a workpiece area;

an electron source for generating an electron beam;

a scanning actuator for controlling the relative movement between the electron beam and the workpiece, the scanning actuator being controllable for directing the electron beam at a series of spaced apart points within the workpiece area;

a first processing system for generating crystallographic data based upon electron diffraction from the workpiece;

a second processing system configured for determining whether sufficient data have been acquired to characterize the workpiece area; and

a controller for controlling the scanning actuator to space the points apart such that acquired data is representative of a different grain within the workpiece.

8. The system of claim 6, the crystallographic characterization subsystem further comprising:

a workpiece holder for holding a crystalline workpiece;

a first ion source for generating a first ion beam;

a scanning actuator for controlling the relative movement between the first ion beam and the crystalline workpiece, the scanning actuator being controllable for directing the first ion beam at desired areas of the crystalline workpiece;

an electron detector for detecting secondary electrons emitted from the crystalline workpiece;

a first processing system for creating a contrast intensity image based upon secondary electron emissions from the crystalline workpiece;

a second processing system programmed to provide crystallographic information based on the contrast image intensity data; and

a controller for controlling the scanning actuator for scanning the first ion beam.

9. The system of claim 8, further comprising a second ion source for generating a second ion beam, the second ion source controllable by the scanning actuator.

10. The system of claim 6, the crystallographic characterization system further comprising:

a sample holder for holding a crystalline sample;

a first ion source for generating a first ion beam;

an electron source for generating an electron beam;

a scanning actuator for controlling the relative movement between the first ion beam, the electron beam, and the crystalline sample, the scanning actuator being controllable for directing the

first ion beam at desired areas of the crystalline sample and for directing the electron beam at a series of points within the sample area;

an electron detector for detecting secondary electron emissions from the crystalline sample;

a first processing system for creating a contrast intensity image based upon secondary electron emissions from the crystalline sample and generating crystallographic data based upon electron diffraction from the crystalline sample;

a second processing system programmed to provide crystallographic information based on the contrast image intensity data and configured for determining whether sufficient data have been acquired to characterize the sample area; and

a controller for controlling the scanning actuator to direct the first ion beam at desired areas such that each ion channeling image is representative of channeling directions within the crystalline sample and to space the points apart such that acquired data is representative of a different grains within the crystalline sample.

11. A method for monitoring and controlling a fabrication process comprising:

coupling a plurality of fabrication subsystems that perform respective fabrication steps on a workpiece;

acquiring crystallographic orientation, grain size, or grain morphology from the workpiece after the workpiece undergoes a fabrication step by at least a first subsystem; and

providing information to one or more of the fabrication subsystems for modifying parameters associated with the respective fabrication steps.

12. The method of claim 11, further comprising providing, in a feed back path, information generated in the crystallographic analysis subsystem to at least the first subsystem at least the first subsystem.

13. The method of claim 11, further comprising providing, in a feed-forward path, information generated in the crystallographic analysis subsystem to at least a second subsystem that performs a fabrication step after the first subsystem and after crystallographic analysis of the workpiece.

14. The method of claim 11, further comprising characterizing a roughness of a workpiece.

15. The method of claim 14, further comprising:

measuring the reflectivity in different regions of the workpiece; and

generating a roughness characteristic based on the measured reflectivity.

16. The method of claim 11, further comprising characterizing a reflectivity of a workpiece.

17. The method of claim 11, further comprising characterizing a crystallography of a workpiece.

18. The method of claim 17, further comprising:

providing a workpiece holder for holding the workpiece for characterization of a workpiece area;

generating an electron beam;

controlling the relative movement between the electron beam and the workpiece for directing the electron beam at a series of spaced apart points within the workpiece area;

generating crystallographic data based upon electron diffraction from the workpiece;

determining whether sufficient data have been acquired to characterize the workpiece area; and

controlling the scanning actuator to space the points apart such that acquired data is representative of a different grain within the workpiece.

19. The method of claim 17, further comprising:

providing a workpiece holder for holding a crystalline workpiece;

generating a first ion beam;

controlling the relative movement between the first ion beam and the crystalline workpiece for directing the first ion beam at desired areas of the crystalline workpiece;

detecting secondary electrons emitted from the crystalline workpiece;

creating a contrast intensity image based upon secondary electron emissions from the crystalline workpiece;

providing crystallographic information based on the contrast image intensity data; and

controlling the scanning actuator for scanning the first ion beam.

20. The method of claim 17, further comprising:

generating a second ion beam; and

controlling the relative movement between the first ion beam, the second ion beam and the crystalline workpiece for directing the second ion beam at desired areas of the crystalline workpiece.

21. The method of claim 17, the crystallographic characterization system further comprising:

providing a sample holder for holding a crystalline sample;

generating a first ion beam;

generating an electron beam;

controlling the relative movement between the first ion beam, the electron beam, and the crystalline sample, the scanning actuator being controllable for directing the first ion beam at desired areas of the crystalline sample and for directing the electron beam at a series of points within the sample area;

detecting secondary electron emissions from the crystalline sample;

creating a contrast intensity image based upon secondary electron emissions from the crystalline sample and generating crystallographic data based upon electron diffraction from the crystalline sample;

providing crystallographic information based on the contrast image intensity data and configured for determining whether sufficient data have been acquired to characterize the sample area; and

controlling the scanning actuator to direct the first ion beam at desired areas such that each

ion channeling image is representative of channeling directions within the crystalline sample and to space the points apart such that acquired data is representative of a different grains within the crystalline sample.

IX. APPENDIX B - EVIDENCE

The evidence in this appendix includes a U.S. Patent to Manada and a U.S. Patent to Adams. Manada was entered in the record by the Examiner with the first Examiner's Office Action. Adams was entered in to the record by the Examiner with the Final Rejection.

X. RELATED PROCEEDINGS APPENDIX

NONE

US005463977A

United States Patent [19]

Manada et al.

[11] Patent Number: 5,463,977

[45] Date of Patent: Nov. 7, 1995

[54] **METHOD OF AND APPARATUS FOR EPITAXIALLY GROWING CHEMICAL COMPOUND CRYSTAL**

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[21] Appl. No.: **91,747**

[22] Filed: **Jul. 15, 1993**

Related U.S. Application Data

[60] Continuation of Ser. No. 799,132, Nov. 27, 1991, abandoned, which is a division of Ser. No. 617,554, Nov. 26, 1990, abandoned.

[30] **Foreign Application Priority Data**

Nov. 24, 1989 [JP] Japan 1-305873

[51] **Int. Cl.**[6] **C30B 25/16**

[52] **U.S. Cl.** **117/85**; 117/86; 117/89; 117/105; 117/108

[58] **Field of Search** 117/85, 86, 108, 117/89, 105; 422/105

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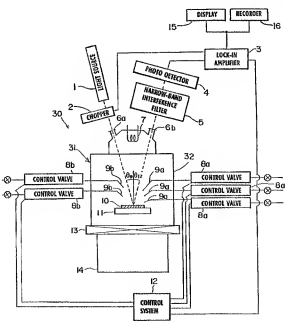
Attorney, Agent, or Firm—Nixon & Vanderhye

[57] **ABSTRACT**

In a method of and an apparatus for epitaxially growing a chemical-compound crystal, a plurality of raw-material gasses are alternately introduced into a closed chamber of a crystal growing device to grow the crystal placed within the closed chamber. At growing of the crystal, a light from a light source is emitted to a crystal growing film of the crystal. Intensity of a light reflected from the crystal growing film and received by a photo detector is measured. Charge amounts of the respective raw-material gasses are controlled by a control system on the basis of a change in the reflected-light intensity, thereby controlling a growing rate of the growing film.

6 Claims, 6 Drawing Sheets



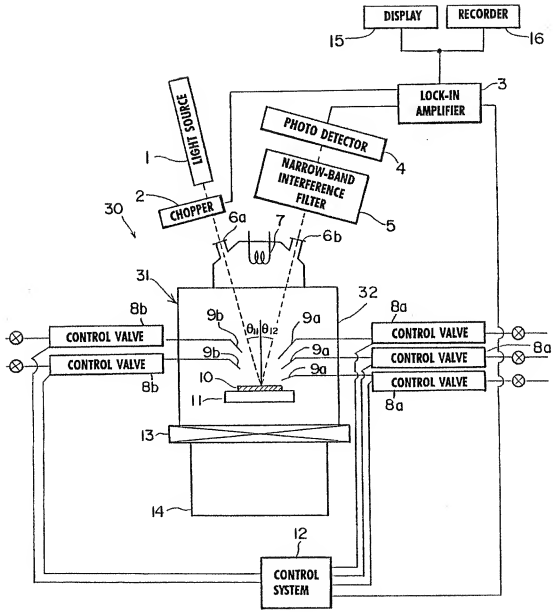

DISPLAY
RECORDER
15
16
LOCK-IN AMPLIFIER
3
PHOTO DETECTOR
4
NARROW-BAND INTERFERENCE FILTER
5
LIGHT SOURCE
1
CHOPPER
2
30
6a
7
6b
31
32
8b
8a
9b
9a
θ_{11}
θ_{12}
CONTROL VALVE
10
11
13
14
12
CONTROL SYSTEM

FIG. 1

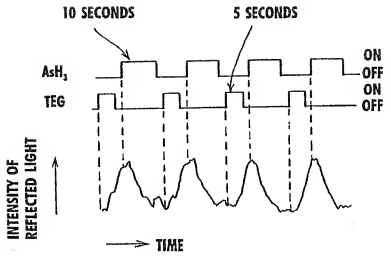

10 SECONDS
5 SECONDS
AsH_3
TEG
ON
OFF
ON
OFF
INTENSITY OF REFLECTED LIGHT
TIME

FIG. 2

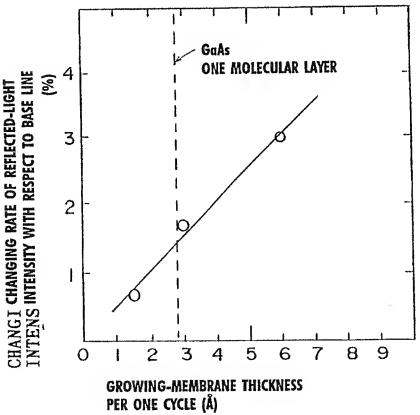

GaAs
ONE MOLECULAR LAYER
CHANGE INTENSITY CHANGING RATE OF REFLECTED-LIGHT INTENSITY WITH RESPECT TO BASE LINE (%)
0
1
2
3
4
5
6
7
8
9
GROWING-MEMBRANE THICKNESS PER ONE CYCLE (Å)

FIG. 3

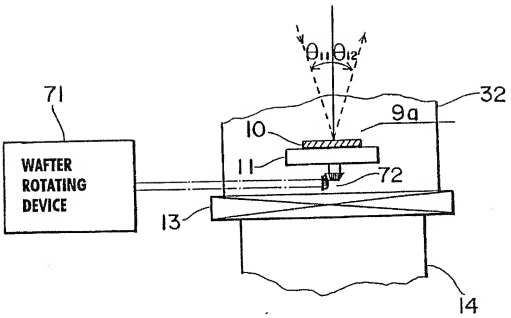


FIG. 4

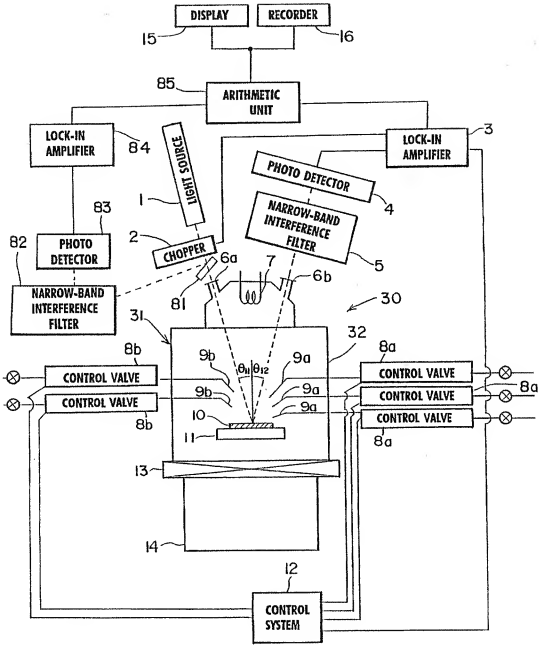

DISPLAY
RECORDER
15
16
85
ARITHMETIC UNIT
LOCK-IN AMPLIFIER
84
LIGHT SOURCE
LOCK-IN AMPLIFIER
3
PHOTO DETECTOR
4
NARROW-BAND INTERFERENCE FILTER
5
1
83
2
CHOPPER
82
PHOTO DETECTOR
6a
7
6b
81
30
NARROW-BAND INTERFERENCE FILTER
31
32
8b
8a
9b
9a
θ11
θ12
CONTROL VALVE
CONTROL VALVE
CONTROL VALVE
CONTROL VALVE
CONTROL VALVE
8a
10
11
13
14
12
CONTROL SYSTEM

FIG. 5

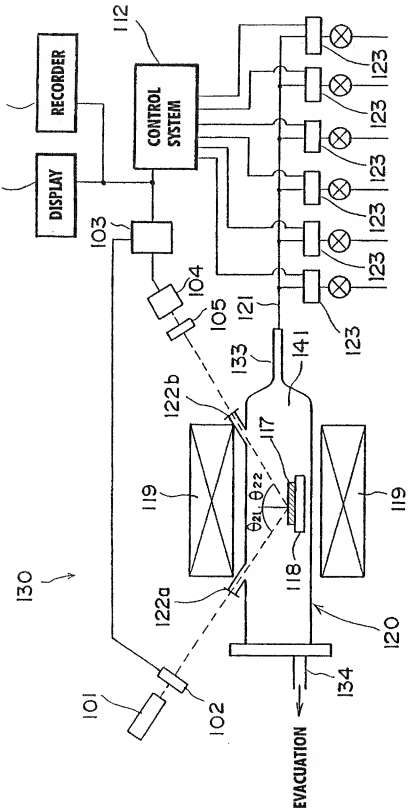

RECORDER
DISPLAY
CONTROL SYSTEM
EVACUATION
112
116
115
103
104
105
121
123
133
141
122b
117
119
θ21
θ22
118
122a
120
134
101
102
130

FIG. 6

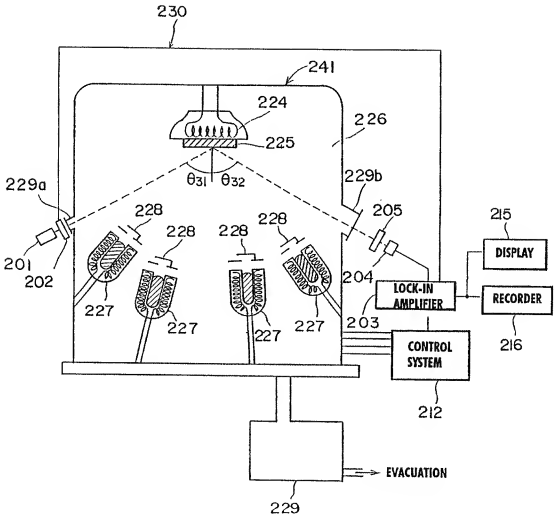

230
241
224
225
226
229a
229b
θ31
θ32
205
215
DISPLAY
228
228
228
228
201
202
204
LOCK-IN AMPLIFIER
RECORDER
227
227
227
227
203
CONTROL SYSTEM
216
212
EVACUATION
229

FIG. 7

METHOD OF AND APPARATUS FOR EPITAXIALLY GROWING CHEMICAL COMPOUND CRYSTAL

This is a continuation of application Ser. No. 07/799,132, filed Nov. 27, 1991, now abandoned, which is a division of application Ser. No. 07/617,554, filed Nov. 26, 1990 now abandoned.

BACKGROUND OF THE INVENTION

The present invention relates to methods of and apparatuses for epitaxially growing a chemical-compound crystal, in which a plurality of raw-material gasses are alternately introduced into a closed chamber of a crystal growing device to grow the crystal and, more particularly, to a method of and an apparatus for epitaxially growing a chemical-compound crystal, in which a growing rate of a growing film of the chemical-compound crystal is controlled at growing of the crystal.

In an epitaxial growing method for a chemical-compound crystal, it is very important to control a growing rate of a growing film of the crystal at growing of the crystal. Conventionally, in a molecular-beam epitaxial growing method and a migration enhanced epitaxial growing method, vibration of a reflection high-energy electron diffraction (hereinafter referred to as "RHEED") is utilized to control the growing rate of the growing film of the order of a monomolecular layer. This is reported in the paper, Jpn. J. Appl. Phys. Vol 23, No. 9 PPL 657–L659 (1984); T. Sakamoto, et al., for example.

In practice, the crystal is grown while rotating a crystal substrate, in order to secure uniformity in a plane of the crystal film.

In the case where the crystal is grown while rotating the crystal substrate as described above, a direction of a crystal axis and a direction of the high-energy electron beam change due to the rotation of the crystal substrate. Thus, it is practically impossible to control the thickness of the growing film of the crystal by the RHEED.

In the existing circumstances, accordingly, the thickness of the growing film of the crystal is controlled by the same conditions as growing conditions which are obtained when the crystal substrate is fixed against rotation. For this reason, it is required to regulate or control charge amounts of the respective raw-material gasses into the crystal growing device by crucibles or the like which are controlled in temperature extremely precisely.

Further, there is the following problems. That is, influence of emission of the high-energy electron beam to the surface of the growing film gives rise to defects in the crystal. Because of the use of the electron beam, the interior of the crystal growing device must be maintained at super high vacuum.

SUMMARY OF THE INVENTION

It is an object of the invention to provide a method of and an apparatus for epitaxially growing a chemical-compound crystal, in which it is possible to secure uniformity in a plane of a crystal film of the crystal, in which any no bad influence is exerted on the crystal at growing of the crystal, and which are applicable to a wide range of pressures without limitation to super high vacuum though it is of course that the method and the apparatus can be applied to the super high vacuum.

According to the invention, there is provided a method of epitaxially growing a chemical-compound crystal, comprising the steps of:

placing the chemical-compound crystal within a closed chamber of a crystal growing device;

alternately introducing a plurality of raw-material gasses into the closed chamber to grow the crystal;

at growing of the crystal, emitting a light to a crystal growing film of the crystal from a predetermined direction;

measuring intensity of a light reflected from the crystal growing film; and

controlling charge amounts of the respective raw-material gasses on the basis of a change in the reflected-light intensity, thereby controlling a growing rate of the growing film.

According to the invention, there is further provided an apparatus for epitaxially growing a chemical-compound crystal, comprising:

a crystal growing device having defined therein a closed chamber, the chemical-compound crystal being placed within the closed chamber;

means for alternately introducing a plurality of raw-material gasses into the closed chamber to grow the crystal;

incident window means provided at a periphery of the closed chamber;

light-source means arranged on the outside of the closed chamber in facing relating to the incident window means, for emitting a light to a crystal growing film of the crystal through the incident window means;

extracting window means provided at the periphery of the closed chamber;

light-receiving means arranged on the outside of the closed chamber in facing relation to the extracting window means, for receiving a light reflected from the crystal growing film of the crystal through the extracting window means;

means for measuring intensity of the reflected light; and

control means for controlling charge amounts of the respective raw-material gasses on the basis of a change in the reflected-light intensity, thereby controlling a growing rate of the growing film of the crystal.

With the above arrangement of the invention, since the intensity of the light reflected from the growing film is measured, any no bad influence is exerted on the inside of the crystal at growing of the crystal.

Further, since the light is used, it is not required to retain or maintain the interior of the crystal growing device at super high vacuum unlike the conventional RHEED, though it is of course that the interior of the crystal growing device may be maintained at the super high vacuum. Since the light is emitted to the crystal growing film from the outside of the closed chamber, pressure within the closed chamber does not become an issue, so that it is possible to apply the method and apparatus to a wide range of pressures including the normal pressure or pressure higher than the same.

Furthermore, since the conventional RHEED is due to electron-beam diffraction, it is necessary to align the direction of the crystal axis and the direction of the high-energy electron beam with each other, so that it is impossible to grow the crystal while rotating the crystal substrate. In the method and the apparatus according to the invention, however, since the change in the reflected-light intensity has no relation to the crystal axis, the crystal substrate may be or may not be rotated at growing of the crystal. Thus, it is possible to secure uniformity in the plane of the crystal-film.

As described above, according to the method and the apparatus of the invention, the use of the light, which is low in cost, enables the growing rate of the growing film to easily be controlled at accuracy equal to or lower than the monomolecular layer.

DETAILED DESCRIPTION OF THE DRAWINGS

FIG. **1** is a schematic view of an epitaxial growing apparatus for carrying out an epitaxial growing method for a chemical-compound crystal, according to a first embodiment of the invention, the epitaxial growing apparatus comprising a GaAs molecular-layer epitaxial growing device;

FIG. **2** is a view showing a change in reflected-light intensity observed at crystal growing of GaAs by the use of the epitaxial growing apparatus illustrated in FIG. **1**;

FIG. **3** is a graphical representation of a mutual relation between a changing rate of the reflected-light intensity and the growing-film thickness per one cycle;

FIG. **4** is a schematic fragmentary view of a first modification of the epitaxial growing apparatus illustrated in FIG. **1**;

FIG. **5** is a schematic view of a second modification of the epitaxial growing apparatus illustrated in FIG. **1**;

FIG. **6** is a schematic view of an epitaxial growing apparatus for carrying out the epitaxial growing method for the chemical-compound crystal, according to a second embodiment of the invention; and

FIG. **7** is a schematic view of an epitaxial growing apparatus for carrying out the epitaxial growing method for the chemical-compound crystal, according to a third embodiment of the invention, the epitaxial growing apparatus comprising a molecular-beam epitaxial device.

DESCRIPTION OF THE EMBODIMENTS

Referring first to FIG. **1**, there is shown an epitaxial growing apparatus which comprises a GaAs molecular-layer epitaxial growing device **30**. The epitaxial growing device **30** is reported in a paper [J. Nishizawa, H. Abe and T. Kurabayasi; J. Electrochem. Soc. 132 (1985) 1197–1200] written by Junichi Nishizawa et el, for example.

As shown in FIG. **1**, the GaAs molecular-layer epitaxial growing device **30** comprises a hermetic or closed container **31** in which a closed crystal growing chamber **32** is defined. For example, pressure within the crystal growing chamber **32** is evacuated, for the first time, to 10^{-6} to 10^{-11} Torr and, subsequently, chemical-compound gas that is a raw material is introduced into the crystal growing chamber **32** at 10^{-1} to 10^{-7} Tort with predetermined sequence while the crystal growing chamber **32** is evacuated. A gate valve **13** is arranged at a location below the closed container **31**. A vacuum evacuation device **14** such as a turbo molecular pump or the like is arranged at a location below the gate valve **13**. The crystal growing chamber **32** is arranged at a location above the gate valve **18**. A GaAs single-crystal substrate susceptor **11** and a GaAs single-crystal substrate **10** are accommodated within the crystal growing chamber **82**. Further, within the crystal growing chamber **82**, a plurality of raw-material-gas introducing nozzles **9***a* and a plurality of impurity-gas introducing nozzles **9***b* are arranged in opposed relation to each other, in facing relation to the GaAs single-crystal substrate **10**. The raw-material-gas introducing nozzles **9***a* and the impurity-gas introducing nozzles **9***b* are provided with their respective control valves **8***a* and **8***b*. These control valves **8***a* and **8***b* are connected to a control system **12**.

Furthermore, a GaAs single-crystal substrate heating lamp **7** is arranged at an upper portion of the crystal growing chamber **82**. An incident window **6***a* and an extracting window **6***b*, through which a light can pass, are provided at a periphery of the crystal growing chamber **82** and are arranged respectively on both sides of the GaAs single-crystal substrate heating lamp **7**. The incident window **6***a* and the extracting window **6***b* are arranged as follows. That is, the incident window **6***a* and the extracting window **6***b* are set in angle such that the light passing through the incident window **6***a* and the extracting window **6***b* is brought to a predetermined incident angle θ_{11} and a predetermined reflecting angle θ_{12} with respect to the GaAs single-crystal substrate **10**.

That is, the arrangement is such that the light from a light source **1** is emitted to a growing film of the GaAs single-crystal substrate **10** from a predetermined direction. The light source **1** is arranged on the outside of the incident window **6***a* in facing relation thereto. The light source **1** is a source of a parallel beam, and is selected from a He-Ne laser, a semiconductor laser, a light-emitting diode, a mercury lamp and an argon ion laser. A chopper **2** for chopping the light is arranged between the incident window **6***a* and the light source **1**. Moreover, a photo detector **4** for the light reflected from the GaAs single crystal substrate **10** is arranged on the outside of the extracting window **6***b* in facing relation thereto. A narrow-band interference filter **5** for removing a stray light from the reflected light is arranged between the extracting window **6***b* and the photo detector **4**.

The chopper **2** and the photo detector **4** are connected to a lock-in amplifier **3**. The lock-in amplifier **3** is provided with a display **15** and a recorder **16**. The lock-in amplifier **3** is connected to the control system **12**.

The epitaxial growing method for the chemical-compound crystal, according to the first embodiment of the invention, is carried out by the use of the GaAs molecular-layer epitaxial growing device **30** as follows.

First, the control valves **8***a* and **8***b* for the impurity gas and the raw-material gas are controlled by the control system **12**. By doing so, the raw-material gas containing Ga and the raw-material gas containing As are alternately introduced into the crystal growing chamber **32** and are directed onto the GaAs single-crystal substrate **10** which is controlled in temperature by the GaAs single-crystal substrate heating lamp **7**, so that GaAs is epitaxially grown.

The reflected-light intensity at the GaAs crystal surface is measured as follows. That is, first, the light from the light source **1** arranged on the outside of the closed container **31** is chopped by the chopper **2**. The light chopped is emitted to the GaAs single crystal through the incident window **6***a*. The light reflected is detected by the photo detector **4** such as a Si photodiode or the like, through the other extracting window **6***b*. The filter **5** is arranged between the photo detector **4** and the extracting window **6***b*, and removes the stray light from the light incident upon the photo detector **4** before the detection. A detecting output from the photo detector **4** is processed in signal by the lock-in amplifier **3**. Subsequently, outputs are successively issued from the lock-in amplifier **3** to the control system **12**, the display **15** and the recorder **16**.

FIG. **2** shows introducing sequence of the raw-material gasses and the change in the reflected-light intensity observed when GaAs is epitaxially grown by the GaAs molecular-layer epitaxial growing device **30** illustrated in FIG. **1**.

As shown in FIG. **2**, it will be seen that the reflected-light intensity increases with introduction of triethylgallium (TEG) which is used as the raw-material gas containing Ca, while the reflected-light intensity decreases with introduction of arsine (AsH_3) which is used as the raw-material gas containing As.

Further, FIG. **3** shows the mutual relation between the growing-film thickness per one cycle and a changing rate of the reflected-light intensity with respect to a base line which is observed when GaAs is epitaxially grown by the GaAs molecular-layer epitaxial growing device **30** illustrated in FIG. **1**.

The above-described mutual relation between the growing-film thickness per one cycle and the changing rate of the reflected-light intensity is utilized to measure a change of the reflected-light intensity illustrated in FIG. **2**. On the basis of the measurement value, the introducing pressure and the introducing time of the raw-material gasses are regulated or controlled. The growing rate of the growing film is controlled at accuracy equal to or lower than the monomolecular layer during growth of the crystal.

For example, in FIG. **1**, the growing rate of the growing film is controlled at accuracy equal to or lower than the monomolecular layer such that the output from the lock-in amplifier **3** is inputted to the control system **12**, the desirable change in intensity corresponding to the growing-film thickness per one cycle is beforehand stored in the control system **12**, and introduction of the raw-material gasses stops when a change in the output from the lock-in amplifier **3** reaches a value of the stored intensity change. Thus, it is possible to produce a desirable growing-film thickness.

In the apparatus illustrated in FIG. **1**, the substrate susceptor **11** is fixedly mounted to the periphery of the crystal growing chamber **32**. As illustrated in FIG. **4** which shows a first modification of the epitaxial growing apparatus, however, the substrate susceptor **11** may be connected to a wafer rotating device **71** through a gear train **72**, so that the substrate **10** on the substrate susceptor **11** can be rotated about the vertical axis by the wafer rotating device **71**. In the first modification illustrated in FIG. **4**, even if the crystal is grown while rotating the crystal substrate, a direction of the light from the light source **1** and a direction of the light reflected from the substrate **10** do not change with respect to the vertical crystal axis. Thus, it is possible to control the thickness of the growing film of the crystal accurately.

FIG. **5** shows a second modification of the apparatus illustrated in FIG. **1**. In the second modification illustrated FIG. **5**, a half-transparent mirror or a half-silvered mirror **81** is arranged between the chopper **2** and the incident window **6***a*. A photo detector **83** is arranged in facing relation to the half-silvered mirror **81**, and a narrow-band interference filter **82** is arranged between the half-silvered mirror **81** and the photo detector **83**. The photo detector **83** is connected to a lock-in amplifier **84**. The lock-in amplifier **84** and the lock-in amplifier **3** are connected to an arithmetic unit **85**. The display **15** and the recorder **16** are connected to the arithmetic unit **85**.

In the epitaxial growing apparatus illustrated in FIG. **5**, a part of the light emitted from the light source **1** is reflected by the half-silvered mirror **81**. The remaining light passes through the half-silvered mirror **81** and is incident upon the substrate **10** through the incident window **6***a*. That is, the light part is extracted from the light incident upon the substrate **10**. The light part serves as a reference light. The light part reflected by the half-silvered mirror **81** is incident upon the photo detector **83** through the narrow-band interference filter **82**. The lock-in amplifier **84** connected to the photo detector **83** issues an output on the basis of the light incident upon the photo detector **83**. At the arithmetic unit **85**, the reference light is compared with The reflected light incident upon the photo detector **4**, in order to reduce instability in output from the light source **1**. Thus, it is possible to control the growing rate at further high accuracy.

Referring next to FIG. **6**, there is shown an epitaxial growing apparatus according to a second embodiment of the invention. The epitaxial growing apparatus comprises a crystal growing device **130** which is one selected from a MO-CVD (metal organic-chemical vapor deposition) device, a chloride-method gas-phase growing device and a hydride epitaxial growing device. These devices are different from each other in gasses utilized. That is, $AsCl_3$ and H_2 are used in the chloride-method gas-phase growing device. HCl, H_2, As_2 and As_4 are utilized in the hydride epitaxial growing device. $Ga(CH_3)_3$, AsH_3 and H_2 are employed in the MO-CVD device.

As shown in FIG. **6**, the crystal growing device **130** comprises a cylindrical quartz reaction tube **120** in which a closed chamber **141** is defined. Pressure within the closed chamber **141** is maintained at the normal pressure or atmospheric pressure to 10^{-2} Tort, for example. The quartz reaction tube **120** has its one end provided with a gas introduction port **133**, and the other end provided with a gas discharge port **134**. Connected to the gas introduction port **133** is a supply nozzle **121** for gasses including GaAs crystal-growing raw material gasses and dopant. A plurality of control valves **123** are connected to the gas supply nozzle **121** and are also connected to a control system **112**.

Further, a quartz susceptor **118** and a GaAs single-crystal substrate **117** are accommodated in the quartz reaction tube **120**, that is, are placed within the closed chamber **141**. A cylindrical heater **119** is arranged about the quartz reaction tube **120**. An incident window **122***a* and an extraction window **122***b*, through which a light can pass, are arranged at their respective locations on both sides of the heater **119** and above the same. The incident window **122***a* and the extracting window **122***b* are arranged as follows. That is, both the windows **122***a* and **122***b* are set in angle such that the light passing through the incident window **122***a* and the extracting window **122***b* is brought to a predetermined incident angle θ_{21} and a predetermined reflecting angle θ_{22} with respect to the GaAs single-crystal substrate **117**.

That is, the arrangement is such that the light from a light source **101** is emitted to a growing film of the GaAs single-crystal substrate **117** from a predetermined direction. The light source **101** is arranged on the outside of the incident window **122***a* in facing relation thereto. The light source **101** is a source of a parallel beam, and is selected from a He-Ne laser, a semiconductor laser, a light-emitting diode, a mercury lamp and an argon ion laser. A chopper **102** for chopping the light from the light source **101** is arranged between the incident window **122***a* and the light source **101**. Moreover, a photo detector **104** for a light reflected from the substrate **117** is arranged on the outside of the extracting window **122***b* in facing relation thereto. A narrow-band interference filter **105** for removing a stray light from the reflected light is arranged between the extracting window **122***b* and the photo detector **104**.

The chopper **102** and the photo detector **104** are connected to a lock-in amplifier **103**. The lock-in amplifier **103** is provided with a display **115** and a recorder **116**. The lock-in amplifier **103** is connected to the control system **112**.

The crystal growing apparatus constructed as described

above is used to carry out the epitaxial growing method for the chemical-compound crystal, according to the second embodiment of the invention, as follows.

First, gas introduction time and evacuation time of the quartz reaction tube **120** are controlled by the control system **112**. The raw-material gas containing Ga and the raw-material gas containing As are alternately supplied onto the GaAs single crystal substrate **117**. In this manner, GaAs is epitaxially grown.

At growth of the crystal, the light from the light source **101** is reflected by the GaAs single-crystal substrate **117**, and intensity of the reflected light is measured by the photo detector **104** such as a Si photodiode or the like. A detection output of the reflected-light intensity from the photo detector **104** is processed in signal by the lock-in amplifier **103**. Subsequently, outputs are successively issued from the lock-in amplifier **103** to the display **115** and the recorder **116**.

Further, the output from the lock-in amplifier **103** is inputted also to the control system **112**. If a change in the reflected-light intensity corresponding to the desirable growing-film thickness per one cycle is reached, the control valve **123** is closed to stop introduction of the GaAs crystal growing raw-material gasses and the gas containing the dopant, thereby controlling the growing rate of the growing-film thickness. Thus, the desirable growing-film thickness is produced.

In connection with the above, the method according to the second embodiment of the invention can equally be applied to gas-phase growth using $GaCl_3$ and AsH_3, chloride-method gas-phase growth due to $Ga—AsCl_3—H_2$, and hydride-method gas-phase growth using $Ga—AsH_3—HCl$.

Although not shown, it is needless to say that the arrangements illustrated in FIGS. **4** and **5** are equally applicable to the arrangement illustrated in FIG. **6**.

Referring next to FIG. **7**, there is shown an epitaxial growing apparatus according to a third embodiment of the invention. The epitaxial growing apparatus comprises a molecular-beam epitaxial growing device **230** which is one of GaAs gas-phase growing devices.

As shown in FIG. **7**, the molecular-beam epitaxial growing device **230** comprises a hermetic or closed container **241** in which a super-high vacuum growing chamber **226** is defined. Pressure within the super-high vacuum chamber **226** is maintained at 10^{-4} to 10^{-11} Tort, for example. A vacuum evacuation device **229** such as an ion pump, a turbo molecular pump or the like is connected to a bottom of the super-high vacuum growing chamber **226**. A substrate heater **224** is arranged at a ceiling within the super-high vacuum growing chamber **226**. A GaAs single-crystal substrate **225** is arranged adjacent the substrate heater **224** and facing downwardly.

Further, a plurality of raw-material heating crucibles **227** are fixedly supported on the bottom within the super-high vacuum growing chamber **226**. A plurality of shutters **228** for controlling supply of molecular beams or raw-material gasses are provided respectively at openings of the respective raw-material heating crucibles **227**. An incident window **229***a* and an extraction window **229***b*, through which a light can pass, are arranged respectively at opposed side walls of the super-high vacuum growing chamber **226**. The incident window **229***a* and the extracting window **229***b* are arranged as follows. That is, the incident window **229***a* and the extracting window **229***b* are set in angle such that the light passing through the incident window **229***a* and the extracting window **229***b* is brought to a predetermined incident angle θ_{31} and a predetermined reflecting angle θ_{32} with respect to the GaAs single-crystal substrate **225**.

That is, the arrangement is such that a light from a light source **201** is emitted to a growing film of the GaAs single-crystal substrate **225** from a predetermined direction. The light source **201** is arranged on the outside of the incident window **229***a* in facing relation thereto. The light source **201** is a source of a parallel beam, and is selected from a He-Ne laser, a semiconductor laser, a light-emitting diode, a mercury lamp and an argon ion laser. A chopper **202** for chopping the light is arranged between the incident window **229***a* and the light source **201**.

Moreover, a photo detector **204** for the light is arranged on the outside of the extracting window **229***b* in facing relation thereto. A narrow-band interference filter **205** for removing a stray light from the light reflected from the substrate **225** is arranged between the extracting window **229***b* and the photo detector **204**. The chopper **202** and the photo detector **204** are connected to a lock-in amplifier **203**. The lock-in amplifier **203** is provided with a display **215** and a recorder **216**. The lock-in amplifier **203** is connected to a control system **212**.

Furthermore, the control system **212** is connected to an electric system for the super-high vacuum growing chamber **226**. The electric system is connected in line to attachment devices such as the shutters **228** and the like which are mounted within the super-high vacuum growing chamber **226**.

The epitaxial growing apparatus is used to carry out the epitaxial growing method for the chemical-compound crystal, according to the third embodiment of the invention, as follows.

First, opening and closing operation of the shutters **228** is controlled by the control system **212**. The raw materials within the respective crucibles **227** are heated respectively by the heaters **227**, and the raw-material gasses are emitted from the crucibles **227** toward the substrate **225** when the shutters **228** are moved to their respective open positions. The raw-material gasses of respective Ga and As are alternately supplied onto the GaAs single-crystal substrate **225**. In this manner, GaAs is epitaxially grown.

The reflected-light intensity is measured as follows. That is, first, the light from the light source **201** arranged on the outside of the molecular-beam epitaxial growing device **230** is chopped by the chopper **202**. Subsequently, the light is applied to the GaAs single-crystal substrate **225** through the incident window **229***a*. The light reflected from the substrate **225** is emitted toward the photo detector **204** through the extracting window **229***b*, and is measured by the photo detector **204** such as a Si photodiode or the like which is arranged on the outside of the molecular-beam epitaxial growing device **230**. At the measurement due to the photo detector **204**, a stray light is attenuated by the narrow-band interference filter **205** which is arranged between the photo detector **204** and the extracting window **229***b*.

A detection output from the photo detector **204** is processed in signal by the lock-in amplifier **203**. Subsequently, outputs are successively issued from the lock-in amplifier **203** to the display **215** and the recorder **216**.

Simultaneously, the output from the lock-in amplifier **203** is also inputted to the control system **212**. The desirable changing rate in the reflected-light intensity corresponding to the growing-film thickness per one cycle is stored in the control system **212**. If a changing rate of the output from the lock-in amplifier **203** reaches a value of the stored desirable changing rate, the shutters **228** are closed.

Although not shown, it is needless to say that the arrange-

ments illustrated in FIGS. **4** and **5** are equally applicable to the arrangement illustrated in FIG. **7**.

As described above, in the first to third embodiments of the invention, the growing rate of the growing-film thickness is controlled during growth of the crystal in the manner mentioned previously. Thus, it is possible to produce a desirable crystal-film thickness at accuracy equal to or lower than a monomolecular layer.

Furthermore, since the conventional RHEED is due to electron-beam diffraction, it is required that the direction of the crystal axis and the direction of the high-energy electron beam are aligned with each other. Thus, it is impossible for the conventional RHEED to grow the crystal while rotating the GaAs single-crystal substrate **10**, **117** or **225**. In the method and the apparatus according to the invention, however, since the change in the reflected-light intensity has no relation to the crystal axis, it is possible to grow the crystal while rotating the GaAs single-crystal substrate **10**, **117** or **225**. Thus, it is possible to secure uniformity in the plane of the crystal film.

What is claimed is:

1. A method of epitaxially growing a chemical-compound crystal in order to generate adsorption and surface reaction per a single molecular layer alternately and independently of each other, said method comprising the steps of:

(a) alternately introducing raw-material gases into a crystal growing vacuum chamber to grow a crystal film monolayer by monolayer by using adsorption and surface reaction of said gases;

(b) during the growth of said crystal, emitting a light onto the growing crystal film of said crystal;

(c) measuring the intensity of a light reflected from the growing crystal film; and

(d) controlling the adsorption amounts of the respective raw-material gases on the growing surface on the basis of a change in the reflected-light intensity, thereby controlling the growth rate of the growing film and the film thickness.

2. The method according to claim **1**, wherein said light emitted on to said growing crystal film is a parallel beam.

3. The method according to claim **2**, wherein said parallel beam is emitted from a semiconductor laser.

4. The method according to claim **2**, wherein said parallel beam is emitted from a light emitting diode.

5. The method according to claim **2**, wherein said parallel beam is emitted from a mercury lamp.

6. The method according to claim **2**, wherein said parallel beam is emitted from an argon laser.

* * * * *

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United States Patent [19]

Adams et al.

[11] Patent Number: 5,466,934

[45] Date of Patent: Nov. 14, 1995

[54] METHOD AND APPARATUS FOR IDENTIFICATION OF CRYSTALLOGRAPHIC DEFECTS

[76] Inventors: **Brent L. Adams**, 3808 Kim La., Gibsonia, Pa. 15044; **David J. Dingley**, 4099 S. 550 East #15F, Salt Lake City, Utah 84107; **David P. Field**, 628 E. 630 North, Pleasant Grove, Utah 84062

[21] Appl. No.: **374,214**

[22] Filed: **Jan. 18, 1995**

[51] **Int. Cl.**[6] **G01N 23/203**

[52] **U.S. Cl.** **250/307**; 250/310

[58] **Field of Search** 250/307, 310

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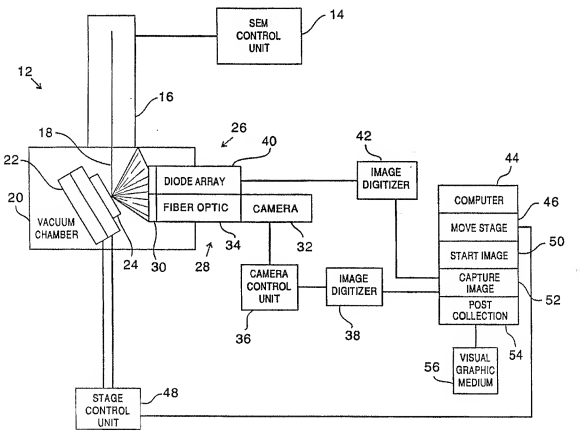
Primary Examiner—Jack I. Berman

Attorney, Agent, or Firm—Lowell W. Gresham; Jordan M. Meschkow

[57] **ABSTRACT**

An imaging apparatus (**10**) includes a scanning electron microscope (**12**) which is controlled to bombard numerous points (**62**) of a material sample (**24**) with an electron beam (**18**). Backscatter diffraction patterns are collected by an image collection system (**26**) which may include both a slower responding video camera (**32**) and a faster responding diode array (**40**). For a baseline point (**62**), an electron backscatter diffraction pattern collected at the video camera (**32**) is analyzed to identify representative pixels which reside along Kikuchi bands (**78**). Backscatter images from subsequent points (**62**) are rapidly compared (**98**) with the baseline to detect changes. When changes are not detected, EBSPs are not analyzed. When changes are detected, EBSPs are analyzed to generate new baselines. The resulting collection of analyzed EBSPs are processed (**104**) to identify microstructure attributes and to characterize defects (**64**).

28 Claims, 4 Drawing Sheets



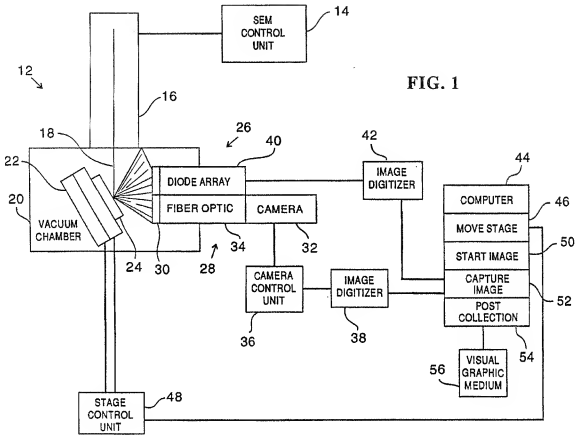

SEM CONTROL UNIT
14
12
16
18
26
40
42
22
DIODE ARRAY
IMAGE DIGITIZER
44
COMPUTER
20
FIBER OPTIC
CAMERA
46
MOVE STAGE
VACUUM CHAMBER
50
START IMAGE
24
30
28
34
32
CAMERA CONTROL UNIT
IMAGE DIGITIZER
CAPTURE IMAGE
52
POST COLLECTION
36
38
54
56
VISUAL GRAPHIC MEDIUM
STAGE CONTROL UNIT
48
10

FIG. 1

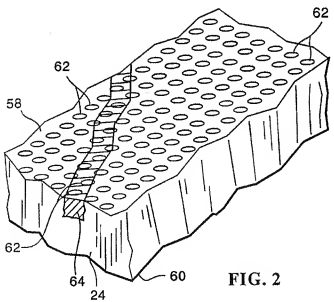
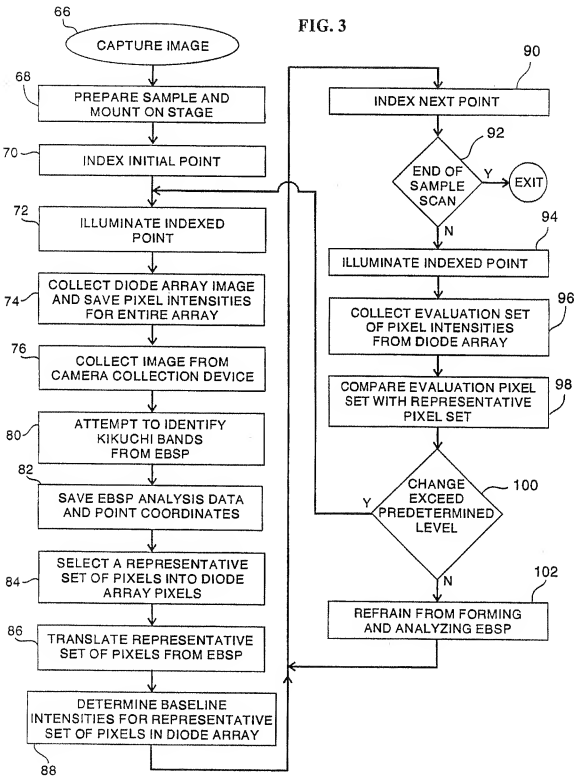

62
62
58
62
64
24
60

FIG. 2

FIG. 3


66
CAPTURE IMAGE
68
PREPARE SAMPLE AND MOUNT ON STAGE
70
INDEX INITIAL POINT
72
ILLUMINATE INDEXED POINT
74
COLLECT DIODE ARRAY IMAGE AND SAVE PIXEL INTENSITIES FOR ENTIRE ARRAY
76
COLLECT IMAGE FROM CAMERA COLLECTION DEVICE
80
ATTEMPT TO IDENTIFY KIKUCHI BANDS FROM EBSP
82
SAVE EBSP ANALYSIS DATA AND POINT COORDINATES
84
SELECT A REPRESENTATIVE SET OF PIXELS INTO DIODE ARRAY PIXELS
86
TRANSLATE REPRESENTATIVE SET OF PIXELS FROM EBSP
88
DETERMINE BASELINE INTENSITIES FOR REPRESENTATIVE SET OF PIXELS IN DIODE ARRAY
90
INDEX NEXT POINT
92
END OF SAMPLE SCAN
Y
EXIT
N
94
ILLUMINATE INDEXED POINT
96
COLLECT EVALUATION SET OF PIXEL INTENSITIES FROM DIODE ARRAY
98
COMPARE EVALUATION PIXEL SET WITH REPRESENTATIVE PIXEL SET
100
CHANGE EXCEED PREDETERMINED LEVEL
Y
N
102
REFRAIN FROM FORMING AND ANALYZING EBSP

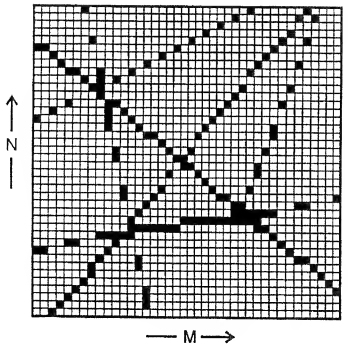


FIG. 4

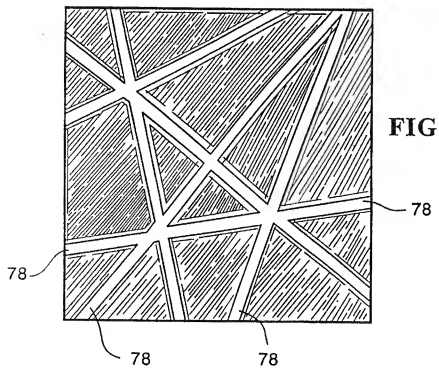


FIG. 5

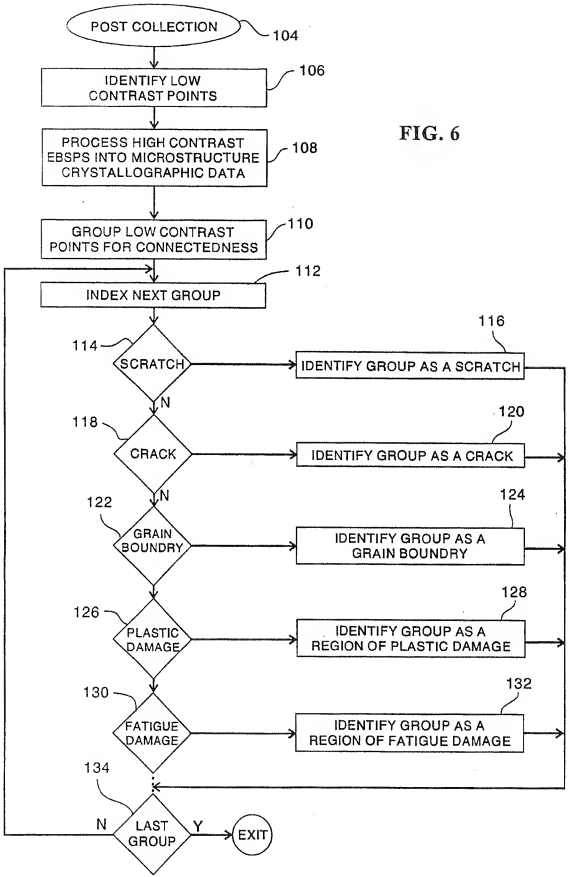

POST COLLECTION
104
IDENTIFY LOW CONTRAST POINTS
106
PROCESS HIGH CONTRAST EBSPS INTO MICROSTRUCTURE CRYSTALLOGRAPHIC DATA
108
GROUP LOW CONTRAST POINTS FOR CONNECTEDNESS
110
INDEX NEXT GROUP
112
114
SCRATCH
116
IDENTIFY GROUP AS A SCRATCH
N
118
CRACK
120
IDENTIFY GROUP AS A CRACK
N
122
GRAIN BOUNDRY
124
IDENTIFY GROUP AS A GRAIN BOUNDRY
126
PLASTIC DAMAGE
128
IDENTIFY GROUP AS A REGION OF PLASTIC DAMAGE
130
FATIGUE DAMAGE
132
IDENTIFY GROUP AS A REGION OF FATIGUE DAMAGE
134
N
LAST GROUP
Y
EXIT

FIG. 6

METHOD AND APPARATUS FOR IDENTIFICATION OF CRYSTALLOGRAPHIC DEFECTS

TECHNICAL FIELD OF THE INVENTION

The present invention relates generally to material sciences. More specifically, the present invention relates to orientation imaging microscopy and to the analysis of electron backscatter diffraction to detect defects in polycrystalline materials.

BACKGROUND OF THE INVENTION

Polycrystalline materials serve as the basic building blocks for a vast assortment of man-made articles. The properties and behaviors of these materials are determined, at least in part, by the size and shape of the constituent crystallites or grains, the orientation of their crystal lattices, and how the grains are placed to fill space. Accordingly, these attributes of the materials microstructure must be determined in order to understand why certain materials behave as they do, to predict how materials will behave, and to alter or otherwise control material forming and processing techniques to improve specific material properties.

Automated orientation imaging microscopy (OIM) has enabled researchers, material processors, and manufacturers to obtain much valuable microstructure information over a relatively large material sample area. Generally, OIM repetitively bombards selected points of a material sample with a beam of electrons. The electrons interact with a small volume of the material sample at the selected points, and backscatter diffraction patterns form on a phosphor screen near the specimen and may be imaged through a video camera. The video images are called electron backscatter diffraction patterns (EBSPs) or backscatter Kikuchi diffraction (BKD) patterns.

Good quality, high contrast, EBSPs include a number of intersecting, relatively high intensity "Kikuchi" bands generally bordered by thin dark lines. The Kikuchi bands result from electrons being diffracted from various planes in the crystal lattice at the point of bombardment. An abundance of microstructure information, including lattice orientation, may be obtained by analyzing the various parameters of the Kikuchi bands. Sophisticated computer-implemented image processing techniques have been developed to analyze Kikuchi bands from EBSPs taken at numerous points on a material sample and to combine this information into OIM maps which describe a wealth of microstructure information.

Unfortunately, each EBSP may include hundreds of thousands of pixels, and tens of thousands of points on a single material sample may be bombarded with an electron beam to produce tens of thousands of EBSPs. Consequently, an immense number of computer operations must be performed to form a single OIM map. Even with very fast computers, the entire process of forming and analyzing each OIM map takes an undesirably long period of time.

A defect in a material is a relatively small region of a material sample which exhibits microstructure properties that differ from the properties exhibited throughout a larger region near the small region. Thus, examples of defects include grain boundaries, cracks, scratches, voids, plastic deformation, fatigue damage, and the like. In materials science and engineering, defects and the microstructure surrounding defects are often of much interest. Electron bombardment at a defect often yields a low quality EBSP

from which Kikuchi bands may be detected with only a low degree of confidence. Consequently, defect information on conventional OIM maps is indirectly inferred from the absence of high confidence microstructure information at particular coordinates on a material sample after an exhaustive and thorough OIM analysis has taken place.

SUMMARY OF THE INVENTION

Accordingly, it is an advantage of the present invention that an improved method and apparatus for identifying crystallographic defects are provided.

Another advantage of the present invention is that defect coordinates are identified without performing a complete microstructure analysis for numerous material sample points that do not reside near a defect.

Another advantage of the present invention is that microstructure information for a large material sample is obtained quickly.

Another advantage of the present invention is that defect information is associated with microstructure attributes in the vicinity of defects.

The above and other advantages of the present invention are carried out in one form by a method of identifying crystallographic defects. The method calls for illuminating a first point on a material sample. A backscatter diffraction image is collected in response to the illumination. A second point is illuminated on the material sample, and a backscatter diffraction image is collected for the second point. The backscatter diffraction images collected for the first and second points are evaluated to determine whether differences between the images exceed a predetermined level. Coordinates of the second point are identified as belonging to a crystallographic defect when the differences exceed the predetermined level.

BRIEF DESCRIPTION OF THE DRAWINGS

A more complete understanding of the present invention may be derived by referring to the detailed description and claims when considered in connection with the Figures, wherein like reference numbers refer to similar items throughout the Figures, and:

FIG. **1** shows a block diagram of a crystallographic defect and orientation imaging apparatus;

FIG. **2** shows a perspective schematic view of an exemplary portion of a polycrystalline material sample which is subjected to electron bombardment in the imaging apparatus;

FIG. **3** shows a flow chart of a capture image process performed by the imaging apparatus;

FIG. **4** shows a schematic representation of an exemplary electron backscatter diffraction image collected through a diode array image collection device of the imaging apparatus;

FIG. **5** shows a schematic representation of an exemplary electron backscatter diffraction pattern (EBSP) obtained through a camera image collection device of the imaging apparatus; and

FIG. **6** shows a flow chart of a post collection process performed by the imaging apparatus.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

FIG. **1** shows a block diagram of a crystallographic defect and orientation imaging apparatus **10**. Imaging apparatus **10**

incorporates a conventional scanning electron microscope (SEM) **12**. SEM **12** includes a SEM control unit **14** which couples to an electron beam generator **16**. Under the direction of control unit **14**, beam generator **16** discharges a focused electron beam **18** in a vacuum chamber **20**. An adjustable stage **22** is mounted in chamber **20** so that a material sample **24** mounted thereon is bombarded or illuminated by electron beam **18**, preferably at an angle of approximately 70° from perpendicular.

An image collection system **26** is positioned within chamber **20** so that a shower of backscatter electrons diffracted from material sample **24** at a high angle from the incident electron beam **18**, desirably at around 110°, falls thereon. Image collection system **26** includes a video camera collection device **28** in which a screen **30** coated with a scintillating material couples to a video camera **32** through a fiber optic bundle **34**. The screen **30** luminesces in correspondence to the pattern of backscatter diffracted electrons falling thereon. This luminescent image is captured by camera **32**, where it is converted to electrical signals, passed through a camera control unit **36**, and converted into digital data in an image digitizer **38**.

In the preferred embodiment, image collection system **26** additionally includes a diode array **40** having MxN closely positioned diodes, where M and N are integer numbers desirably in the range of three to one hundred. Backscatter diffracted electrons falling on diode array **40** excite various ones of the individual diodes to various degrees, causing diode array **40** to generate electrical signals that describe the pattern and intensity of this excitement. These electrical signals are converted into digital data in an image digitizer **42**. Diode array **40** is positioned near phosphor screen **30** so that the geometric relationship therebetween is known. Diode array **40** is included in the preferred embodiment because of its quick response when compared with video camera collection device **28**. However, in an alternate embodiment diode array **40** may be omitted, which causes a slight degradation in the number of images which may be processed in a unit of time when compared to the preferred embodiment.

A computer **44** controls the detection of defects in material sample **24**. Computer **44** is a conventional computer desirably having as much computing power as is economically practical. Computer **44** includes conventional computer components, including a memory (not shown) which stores programming instructions that define processes carried out by imaging apparatus **10** and which stores data generated by the operation of imaging apparatus **10**.

Computer **44** includes a move stage processor block **46** which controls the movement of stage **22** through a stage control unit **48**. Through control of stage **22** and electronic steering of electron beam **18**, a wide area of material sample **24** may be illuminated by electron beam **18** with backscatter diffraction falling upon image collection system **26**. Computer **44** additionally includes a start image processor block **50**, which coordinates image collection with electron beam activation. A capture image processor block **52** performs initial image processing and coordinates with move stage processor **46** and start image processor **50** to index subsequent backscatter diffraction images. Capture image processor **52** receives input data from image digitizers **38** and **42** and is discussed in more detail below in connection with FIG. **3**. A post collection processor block **54** analyzes data collected from numerous backscatter diffraction images taken from numerous points on material sample **24**. Post collection processor **54** is discussed in more detail below in connection with FIG. **6**. The output from capture image processor **52** and/or post collection processor **54** drives a visual graphic medium **56**, such as a video display terminal, printer, or other device which may serve to convey defect and related microstructure information.

Those skilled in the art will appreciate that processors **46**, **50**, **52**, and **54** implemented within computer **44** need not be separate physical entities but are distinguishable logical entities. With the exception of diode array **40**, image digitizer **42**, and processors **52** and **54**, the components of imaging apparatus are conventional in the art of orientation imaging microscopy and need not be discussed in detail.

FIG. **2** shows a perspective schematic view of an exemplary portion of material sample **24**. Material sample **24** has been prepared by making a target surface **58** reasonably parallel to a mounting surface **60**, which contacts stage **22** (see FIG. **1**). In addition, target surface **58** is reasonably polished to remove scratches. FIG. **2** illustrates numerous points **62** upon target surface **58**. Points **62** indicate the locations upon surface **58** that are bombarded with electron beam **18** (see FIG. **1**). Points **62** may or may not be noticeable on target surface **58** after completion of scanning. Some sort of backscatter diffraction image is collected from each point **62**, as discussed below. Each point **62** represents a small volume of material sample **24**, typically around 0.2 μm in diameter. In the preferred embodiment, the field of points may cover a region more than 0,027×0,026 meters. In alternate embodiments, continuous scanning may be performed so that points **62** represent sections of a continuous scan path.

FIG. **2** illustrates points **62** as forming an array with aligned rows and columns, which is a convenient but otherwise unnecessary arrangement for points **62**. The spacing between points **62** may vary from application to application, and is desirably a parameter which may be set by an operator prior to scanning material sample **24**. Typical values range from less than one μm to one hundred μm. For defect detection, step size (i.e. the distance between points **62**) is typically in the lower region of this range so that defects are less likely to be missed. Of course, smaller step size leads to an increased number of images to process for an entire material sample **24**, and rapid image processing becomes all the more important.

In addition, FIG. **2** schematically illustrates an exemplary defect **64**. Defect **64** may represent a crack, scratch, region of plastic deformation, region of fatigue damage, or even a grain boundary. While different types of defects **64** may exhibit different characteristics, they share the characteristic of having different microstructure attributes from surrounding regions. Thus, the coordinates of a region of changed microstructure coincide with the coordinates of a defect, such as a crack. Different ones of points **62** fall inside and outside of defect **64**.

FIG. **3** shows a flow chart of a capture image process **66** performed by imaging apparatus **10** under the control of capture image processor **52** in computer **44**. Generally, process **66** collects data from electron backscatter diffraction obtained at various points **62** located in and near defects **64**.

Process **66** performs a task **68** to prepare material sample **24** as discussed above in connection with FIG. **2** and to mount sample **24** on stage **22** (see FIG. **1**). Task **68** may be performed with human intervention. After task **68**, material sample **24** is not moved relative to stage **22** until scanning is complete so that a common coordinate system is used for all points **62** (see FIG. **2**) which supply data that identify defects, such as defect **64** (see FIG. **2**), and the microstructure of material sample **24** in the vicinity of defect **64**.

After task **68**, a task **70** moves electron beam **18** to an initial point **62** on material sample **24**. By moving electron beam **18** to a point **62**, computer **44** controls stage **22** and the electronic steering of electron beam **18** (see FIG. **1**) so that electron beam illumination will occur at a desired location on material sample **24**. The initial point may be selected to promote any convenient scanning technique. For example, scanning may start in one corner of material sample **24** and illuminate numerous points in a straight line, then numerous parallel lines of points until an entire sample has been scanned. Alternatively, a centrally located point may be first selected and subsequent points may spiral outward therefrom. Desirably, a scanning pattern is selected so that movement of beam **18** between points is quick and so that consecutively scanned points are generally near one another.

After task **70**, a task **72** activates electron beam **18** to illuminate or bombard the current point **62**. In task **72**, the illumination desirably lasts long enough so that an image is obtained from video camera collection device **28** (see FIG. **1**). This duration is longer than necessary to obtain an image from diode array **40** (see FIG. **1**). Thus, in a task **74** an electron backscatter diffraction image is collected from diode array **40**, with pixel intensities from all diodes in the entire array being saved for potential use later.

FIG. **4** shows a schematic representation of an exemplary electron backscatter diffraction image collected through diode array **40**. Shaded grid locations in FIG. **4** indicate diode array pixels (or individual diodes) reporting a greater intensity. Array **40** may describe its image using up to several thousand individual pixels.

Referring back to FIG. **3**, a task **76** collects an electron backscatter diffraction image from video camera collection device **28**. This image forms an electron backscatter diffraction pattern (EBSP), also know as a backscatter Kikuchi diffraction (BKD) pattern. The image from video camera collection device **28** may be described using hundreds of thousands of individual pixels.

FIG. **5** shows a schematic representation of an exemplary electron backscatter diffraction pattern (EBSP) obtained in task **76**. FIG. **5** illustrates Kikuchi bands **78** as being higher intensity stripes of varying width, spacing, and orientation. Kikuchi bands **78** intersect each other at various points and are generally bordered by thin lines of lower intensity.

However, FIG. **5** exaggerates the contrast available in a typical EBSP for the sake of clarity. The contrast of the image collected in task **76** will vary depending upon the microstructure of material sample **24** at the point **62** being illuminated. For example, when the illuminated point **62** resides in a crack or scratch, the backscattering direction for electrons can lead through a longer material path before exiting from material sample **24**. Consequently, few electrons may exit, and portions or all of the resulting image may be entirely dark. For minor cracks on the order of 50 nm or less and other types of defects, the resulting image may simply exhibit less intensity and poorer contrast than that which results from a point **62** remotely located from a defect.

Referring back to FIG. **3**, after task **76** a task **80** attempts to identify the locations of Kikuchi bands **78** (see FIG. **5**). Task **80** uses well known algorithms, such as the Hough transform, to identify Kikuchi bands **78**. In addition, task **80** may employ other well known image enhancing techniques, such as removing a background before attempting to identify Kikuchi bands **78**. Those skilled in the art will understand that task **80** may be a computationally intense operation which requires a significant amount of time to complete. Moreover, depending upon the contrast available in the EBSP, task **80** may not be entirely successful in identifying Kikuchi bands **78**. Thus, locations identified in task **80** may or may not coincide with actual Kikuchi bands **78**, depending upon the contrast of the image collected above in task **76**. Image contrast may be measured and used to indicate the degree of confidence achieved in task **80**.

Next, a task **82** saves the EBSP analysis data and coordinates of the illuminated point **62** for use later by post collection processor **54**. Microstructure and crystallographic data are obtained from these data. Since material sample **24** remains fixed on stage **22**, the microstructure data will be associated with a coordinate system that likewise identifies defects. Thus, defects are linked to the associated defect microstructure through this coordinate system.

After task **82**, a task **84** selects a representative set of pixels from the EBSP data. In the preferred embodiment, the representative set of pixels are aligned relative to one another to extend along the higher intensity regions of the EBSP which are defined by the Kikuchi bands **78** discovered above in task **80**. This pattern of Kikuchi bands identifies the underlying microstructure, even without specifying what that microstructure may be.

After task **84**, a task **86** translates the representative set of pixels discovered by analyzing the EBSP into a set of pixels in diode array **40**. This translation may be performed in response to the geometric relationship between diode array **40** and video camera collection device **28** (see FIG. **1**). Thus, task **86** identifies those pixels in diode array **40** where the images of the Kikuchi bands should be formed. FIG. **4** illustrates the representative set of pixels translated to diode array **40** by shaded grid locations.

Next, a task **88** determines baseline intensities for the representative set of pixels in diode array **40**. These intensities were recorded above in task **74**, along with intensities at pixels which are not included in the representative set of pixels. After task **88**, a task **90** moves beam **18** to the next point on material sample **24**. The movement of beam **18** is performed in accordance with a scanning algorithm, as discussed above in connection with task **70**. After task **90**, a query task **92** determines whether the entire material sample **24** has been scanned yet. When the entire sample has been scanned, program control may exit capture image process **66** and proceed, for example, to a post collection process discussed below in connection with FIG. **6**.

So long as scanning is not yet complete, a task **94** illuminates the selected point **62** on material sample **24** with electron beam **18** (see FIG. **1**). Task **94** needs to activate electron beam **18** for only the short period of time required to collect an image from diode array **40**. A task **96** then collects an evaluation set of pixel intensities from diode array **40**. The evaluation set corresponds to the representative set determined above in task **86**. In other words, the same pixels on diode array **40** where the image of the Kikuchi bands should be formed are evaluated. Other pixels are ignored to reduce processing time.

After task **96**, a task **98** compares the intensities of the evaluation set of pixels with the baseline intensities determined above in task **88**. The comparison performed by task **98** may be performed on a pixel by pixel basis or may be performed by combining pixel intensities and comparing combined results. After task **98**, a query task **100** evaluates the comparison to determine whether the amount of intensity change in the evaluation set of pixels from the baseline intensities exceeds a predetermined level. The predetermined level may be a variable specified by a user. Regard-

less of the level, when the higher intensity Kikuchi band pixels between different points **62** exhibit little or no change, imaging apparatus **10** concludes that the underlying microstructure has not changed, and that no defect is present at the currently imaged point **62**.

As long as no change is detected in task **100**, process **66** refrains from forming and analyzing an EBSP, as indicated in a task **102**, and program control proceeds back to task **90**. At task **90**, imaging apparatus **10** moves beam **18** to the next point and the illumination, collection, and comparison tasks repeat. Program control remains in the programming loop which includes tasks **90**, **92**, **94**, **96**, **98**, **100**, and **102** until task **100** detects a change when comparing evaluation pixels in electron backscatter diffraction images against the representative baseline. No EBSPs are collected, analyzed, or saved, but points **62** are illuminated, images are collected, and evaluation pixels are analyzed very quickly.

When task **100** detects a change in images, program control returns to task **72**, discussed above. The current point **62**, or perhaps another point **62** near the current point **62**, will be illuminated again, this time for a duration sufficient to allow an EBSP to be formed through the use of video camera collection device **28**. The above-discussed process of collecting the EBSP, analyzing the EBSP to identify Kikuchi bands **78**, and determining a new baseline against which to compare future points **62** repeats.

Referring to FIG. **2**, when the scanning of points **62** does not encounter defect **64**, scanning takes place quickly and redundant EBSPs are not analyzed. When defect **64** is encountered, an image change will be detected and an EBSP will be collected and analyzed. If the EBSP has low intensity or poor contrast and Kikuchi bands **78** cannot be accurately identified, the representative set of pixels may have no actual relationship to Kikuchi bands. Consequently, the subsequent point **62** will most likely be evaluated as yielding a changed image from its baseline. The full analysis, the EBSP mode of operation will continue until image quality improves sufficiently to achieve repeatable results between adjacent points **62**.

In the above-discussed alternate embodiment where diode array **40** is omitted, process **66** may be altered from that described above. For example, tasks **74**, **86**, and **88** may be omitted. Instead, the representative set of pixels may be defined for an EBSP collected for each point **62**. While an EBSP is collected through video camera collection device **28** for each point **62**, not all EBSPs are fully analyzed or saved. Analysis may be omitted for EBSPs whose evaluation pixels indicate no significant change from previous EBSPs. This too achieves image processing speed improvements over conventional techniques while simultaneously locating defects.

FIG. **6** shows a flow chart of a post collection process **104** performed by imaging apparatus **10** under the control of post collection processor **54**. Process **104** is performed after material sample **24** has been entirely scanned. However, nothing requires process **104** to be immediately performed after capture image process **66** (see FIG. **3**). Generally, process **104** analyzes the data saved in the numerous iterations of task **82** from process **66** to obtain microstructure and crystallographic data and to classify the defects found through performing process **66**. These data describe EBSPs and related coordinates obtained from points **62** where image changes were detected by process **66**. Capture image process **66** causes such points **62** to generally reside in and around defects.

Process **104** performs a task **106** to identify the points **62** which yielded low contrast EBSPs. The low contrast EBSPs provide poor data from which microstructure and crystallographic data may be derived. Thus, after task **106** a task **108** processes the higher contrast EBSPs into microstructure and crystallographic data using conventional orientation imaging microscopy (OIM) techniques. Such data include lattice orientation data as an example and indicate coordinates on material sample **24** for the various microstructure attributes.

After task **108**, a task **110** groups the points **62** which yielded lower contrast EBSPs for connectedness. In other words, task **110** identifies the points **62** which reside in continuous or near continuous straight or zig-zagged lines or bands. Connected points **62** are grouped together, and non-connected points **62** are grouped separately. Conventional image processing techniques may be employed at task **110**.

Next, a task **112** identifies a next group from the groups identified above in task **110**. For the first iteration of task **112**, a first group is identified. Nothing requires task **110** to identify multiple groups.

After task **112**, a query task **114** examines the coordinates, EBSP data, and nearby microstructure data for the current group of points **62** to determine whether a scratch is indicated. Generally, a scratch may be detected by noticing a complete loss of EBSP (i.e. dark, low contrast images) over one to several microns. The lost EBSP points may be connected in a continuous or near continuous, generally straight line. The region adjacent to a scratch may be associated with a loss in diffraction intensity and small to large lattice rotations. If a scratch is detected, a task **116** identifies the group of points **62** as a scratch. A group of points which have been identified as a scratch may be ignored in subsequent material analyses because it may indicate nothing more than imperfect sample preparation.

When task **114** determines that the group is not a scratch, a query task **118** evaluates the group to determine whether it is a crack. Generally, characteristics of a crack and a scratch are similar, except that a crack may not exhibit the straightness exhibited by a scratch. If a crack is detected, a task **120** identifies the group of points **62** as being a crack.

When task **118** fails to identify the group of points as being a crack, a query task **122** evaluates the group to determine whether it represents a grain boundary. A grain boundary may be recognized as forming a closed figure made from narrow line segments. Inside the closed figure a relative consistent lattice rotation may be observed, and outside the closed figure lattice rotations may substantially differ from the internal lattice rotations. If a grain boundary is detected, a task **124** identifies the group of points **62** as being a grain boundary.

When task **122** fails to identify the group of points as being a grain boundary, a query task **126** determines whether the group of points **62** exhibits characteristics consistent with a region of plastic damage. Plastic damage may be identified by a generally oscillating loss in EBSP intensity and visual diffuseness spread over tens or hundreds of microns. Task **126** may investigate points included in other groups in making its determination. If plastic damage is detected, a task **128** identifies the group of points **62** as being a region of plastic damage.

When task **126** fails to identify the group of points as being a region of plastic damage, a query task **130** determines whether the group of points **62** exhibits characteristics consistent with a region of fatigue damage. Fatigue damage may be identified by a loss of EBSP sharpness but not a complete loss of EBSP. The loss in intensity may be associated with local lattice rotations, may extend in a continu-

ous line across the material sample, and may be only a few microns wide. If fatigue damage is detected, a task **132** identifies the group of points as being a region of fatigue damage.

As indicated by ellipsis, process **104** may inquire further to classify the defect identified by the current group of points. Eventually program control encounters a query task **134**, which is also performed after each of tasks **116**, **120**, **124**, **128**, and **132**. Task **134** determines whether the just-evaluated group was the last group for the material sample. If it was not the last group, program control loops back to task **112** to identify the next group and repeat the classification process. When the last group has been classified, program control may exit process **104**. Although not shown, the microstructure and defect data may be used in any number of ways, including the forming of maps which may be presented at visual graphic medium **56** (see FIG. **1**).

In summary, the present invention provides an improved method and apparatus for identifying crystallographic defects. Defect coordinates are identified without performing an orientation determination for numerous material sample points that do not reside near a defect. Microstructure attribute information for a large material sample is obtained quickly because redundant analyses are avoided. Defect information is associated with microstructure attributes in the vicinity of defects.

The present invention has been described above with reference to preferred embodiments. However, those skilled in the art will recognize that changes and modifications may be made in these preferred embodiments without departing from the scope of the present invention. For example, those skilled in the art can devise alternate processes, task definition, and sequencing than that described herein to achieve substantially the same result. These and other changes and modifications which are obvious to those skilled in the art are intended to be included within the scope of the present invention.

What is claimed is:

1. A method of identifying crystallographic defects comprising the steps of:

a) illuminating a first point on a material sample;

b) collecting a backscatter diffraction image in response to said step a);

c) illuminating a second point on said material sample;

d) collecting a backscatter diffraction image in response to said step c);

e) determining whether change between said images collected in said steps b) and d) exceeds a predetermined level; and

f) identifying coordinates of said second point on said material sample as belonging to a crystallographic defect when said step e) determines that said change between said images exceeds said predetermined level.

2. A method as claimed in claim **1** wherein said step b) comprises the step of attempting to identify Kikuchi bands in an electron backscatter diffraction pattern (EBSP).

3. A method as claimed in claim **1** additionally comprising the step of attempting to identify Kikuchi bands in an electron backscatter diffraction pattern (EBSP) when said step e) determines that change between said images collected in said steps b) and d) exceeds said predetermined level.

4. A method as claimed in claim **3** wherein said attempting to identify step forms said EBSP for a point on said material sample which is proximate said second point.

5. A method as claimed in claim **1** additionally comprising the step of refraining from attempting to identify Kikuchi bands in an electron backscatter diffraction pattern (EBSP) when said step e) determines that change between said images collected in said steps b) and d) does not exceed said predetermined level.

6. A method as claimed in claim **1** additionally comprising the step of positioning first and second image collection devices so that said backscatter diffraction image in said step b) falls upon both of said first and second devices and so that said backscatter diffraction image in said step d) falls upon both of said first and second devices.

7. A method as claimed in claim **6** additionally comprising the step of configuring said first and second image collection devices so that said second image collection device responds to said image more quickly than said first image collection device.

8. A method as claimed in claim **1** wherein:

said steps b) and d) collect said images at one or more image collection devices which represents each image as a multiplicity of pixels; and

said method additionally comprises the step of identifying, in response to said step b), a set of pixels associated with said image collected in said step d) to evaluate in said determination of said step e).

9. A method as claimed in claim **8** additionally comprising the steps of:

g) illuminating a third point on said material sample;

h) collecting a backscatter diffraction image in response to said step g);

i) identifying, when change between said images collected in said steps b) and d) exceeds said predetermined level, a second set of pixels which is associated with said image collected in said step h); and

j) evaluating said second set of pixels associated with said image collected in said step h) to determine whether change between said images collected in said steps d) and h) exceeds said predetermined level, said evaluating step j) occurring when said step e) determines that change between said images collected in said steps b) and d) exceeds said predetermined level.

10. A method as claimed in claim **8** additionally comprising the steps of:

g) illuminating a third point on said material sample;

h) collecting a backscatter diffraction image in response to said step g); and

i) evaluating said set of pixels associated with said image collected in said step h) to determine whether change between said images collected in said steps b) and h) exceeds said predetermined level, said evaluating step i) occurring when said step e) determines that change between said images collected in said steps b) and d) does not exceed said predetermined level.

11. A method as claimed in claim **1** additionally comprising the steps of:

mounting said sample to a stage prior to said step a); and

obtaining crystallographic orientation data for said sample while said sample is mounted to said stage and before said sample is significantly moved relative to said stage.

12. A method as claimed in claim **11** wherein:

said obtaining step comprises the steps of illuminating a third point on said material sample with an electron beam, collecting an electron backscatter diffraction pattern (EBSP) for said third point, and processing said EBSP to identify lattice orientation for said sample at

said point; and

said step e) takes place more quickly than said processing step.

13. A method as claimed in claim 1 wherein:

said material sample has a crack therein; and

said coordinates identified in said step f) coincide with said crack.

14. A method as claimed in claim 1 additionally comprising the step of controlling a visual graphic medium to depict said material sample and said defect.

15. A method of identifying crystallographic defects comprising the steps of:

a) illuminating a first point on a material sample with an electron beam;

b) collecting an electron backscatter diffraction image in response to said step a);

c) forming an electron backscatter diffraction pattern (EBSP) from said electron backscatter diffraction image collected in said step b);

d) identifying a representative set of pixels from said EBSP;

e) illuminating a second point on said material sample with an electron beam;

f) collecting an electron backscatter diffraction image in response to said step e);

g) determining whether intensities at a set of pixels corresponding to said representative set of pixels indicate change between said images collected in said steps b) and f) in excess of a predetermined level; and

h) identifying coordinates of said second point on said material sample as belonging to a crystallographic defect when said step g) determines that said change exceeds said predetermined level.

16. A method as claimed in claim 15 additionally comprising, when change between said images collected in said steps b) and f) exceeds said predetermined level, the steps of:

forming a second electron backscatter diffraction pattern (EBSP); and

identifying a second representative set of pixels from said second EBSP.

17. A method as claimed in claim 16 wherein said forming step forms said second EBSP for a point on said material sample which is proximate said second point.

18. A method as claimed in claim 16 additionally comprising the step of refraining from forming an EBSP when said step g) determines that change between said images collected in said steps b) and f) does not exceed said predetermined level.

19. A method as claimed in claim 15 additionally comprising the step of positioning first and second image collection devices so that said electron backscatter diffraction image in said step b) simultaneously falls upon said first and second devices and so that said electron backscatter diffraction image in said step f) simultaneously falls upon said first and second devices.

20. A method as claimed in claim 19 wherein:

said forming step c) forms said EBSP from said electron backscatter diffraction image collected at said first image collection device;

said determining step g) evaluates an image collected at said second image collection device; and

said second image collection device responds more quickly than said first image collection device.

21. A method as claimed in claim 19 wherein:

said representative set of pixels is associated with said first image collection device; and

said set of pixels which is evaluated in said step g) and which corresponds to said representative set of pixels is associated with said second image collection device.

22. A method as claimed in claim 15 additionally comprising the steps of:

i) illuminating a third point on said material sample with an electron beam;

j) collecting a backscatter diffraction image in response to said step i);

k) identifying, when change between said images collected in said steps b) and f) exceeds said predetermined level, a second representative set of pixels which is associated with said image collected in said step j); and

l) evaluating a set of pixels associated with said image collected in said step j) and corresponding to said second representative set of pixels to determine whether change between said images collected in said steps f) and j) exceeds said predetermined level, said evaluating step l) occurring when said step g) determines that change between said images collected in said steps b) and f) exceeds said predetermined level.

23. A method as claimed in claim 15 additionally comprising the steps of:

i) illuminating a third point on said material sample with an electron beam;

j) collecting a backscatter diffraction image in response to said step i); and

k) evaluating said set of pixels associated with said representative set of pixels to determine whether change between said images collected in said steps b) and j) exceeds said predetermined level, said evaluating step k) occurring when said step g) determines that change between said images collected in said steps b) and f) does not exceed said predetermined level.

24. A crystallographic defect and orientation imaging apparatus comprising:

an electron beam generator;

a stage upon which a material sample may be mounted to receive electron beam illumination from said electron beam generator;

an image collection system positioned to detect electron backscatter diffraction resulting from illuminating points of said stage-mounted material sample with said electron beam; and

image processing means, coupled to said image collection system, for identifying a representative set of pixels from an electron backscatter diffraction image collected for a first point on said stage-mounted material sample and for comparing said representative set of pixels with an evaluation set of pixels taken from an electron backscatter image collected for a second point on said stage-mounted material sample to determine whether said second image has changed from said first image.

25. A crystallographic defect and orientation imaging apparatus as claimed in claim 24 wherein said image collection system comprises:

a first image collector positioned to detect electron backscatter diffraction resulting from illuminating points of said stage-mounted material sample with said electron beam; and

a second image collector positioned to detect electron backscatter diffraction resulting from illuminating points of said stage-mounted material sample with said electron beam.

26. A crystallographic defect and orientation imaging apparatus as claimed in claim **25** wherein:

said representative set of pixels is selected from an image collected at said first image collector; and

said evaluation set of pixels corresponds to said representative set of pixels and is selected from an image collected at said second image collector.

27. A crystallographic defect and orientation imaging apparatus as claimed in claim **25** wherein said second image collector has a quicker response time than said first image collector.

28. A crystallographic defect and orientation imaging apparatus as claimed in claim **24** wherein said image processing means is configured so that:

said evaluation set of pixels is identified in response to said representative set of pixels; and

said image processing means is configured to compare said representative set of pixels with said evaluation set of pixels more quickly than it identifies said representative set of pixels.

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